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MANUAL

OF

TECHNICAL ANALYSIS:

A GUIDE

FOR THE

TESTING AND VALUATION

OF

THE VARIOUS NATURAL AND ARTIFICIAL SUBSTANCES

EMPLOYED IN THE

ARTS AND IN DOMESTIC ECONOMY.

FOUNDED UPON

THE HANDBUCH DER TECHNISCH-CHEMISCHEN UNTERSUCHUNGEN

OF

DR. P. A. BOLLEY,

PROFESSOR OF CHEMISTRY AND TECHNOLOGY IN THE UNIVERSITY OF ARAU.

BY

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PREFACE.

THE preparation of this Manual has been undertaken in the belief, that the increasing recognition of the value of chemical data for commercial and technical purposes, renders it desirable that the capability of executing chemical analyses and examinations of a certain class, should be a more frequent qualification than hitherto.

It is believed, too, that there are a great number of questions of considerable technical importance, for the determination of which it is not at all necessary that the operator should be possessed of any greater chemical knowledge than may be assumed to belong to the occupation in which he is habitually engaged.

It is not at all my intention, by these remarks, to question the value of sound scientific training, or to imply that a thorough acquaintance with physical science would not be very desirable in many cases where it is commonly wanting. I merely propose to point out, that, even in the absence of such qualifications, there are many well-defined lines of analytical inquiry, which may be advantageously pursued by persons, engaged in business, who may be unable to avail themselves of the very scanty means that exist in this country of acquiring really practical and useful scientific knowledge.

It is to serve as a guide in such cases that this Manual is intended. For the general plan of it, I am indebted to the "Handbuch der technisch-chemischen Untersuchungen" by Dr. Bolley of Arau. In the first instance a translation of

that work was contemplated, but afterwards it appeared advisable to make very considerable alterations and additions. Chapters I. II. VIII. IX. and X. have been entirely re-written, and much new matter introduced into the others. It is with great satisfaction in this respect, that I am able to state, that these alterations have met with the entire approval of Dr. Bolley, who in a letter to me says,—"The "very essential alterations in the arrangement and contents "of the book were soon evident to me. I consider them "much to its advantage, and perceive from the general "treatment of the subject, that the book has fallen into just "the right hands to be presented to English readers in an "appropriate manner."

In conclusion, I may remark that in a work of this kind, which cannot lay claim to originality other than that which may appertain to the adaptation and arrangement of existing material, there will of course be much that is borrowed. Supposing this to be generally understood, I have limited myself to the endeavour to describe briefly and simply the methods of carrying out different examinations, and have availed myself of what has been published on the subject by various chemists, without especial reference to the authors. I have to express my thanks to Mr. Phillips of the Excise, for tables of the relation between the density of spirit and the per-centage of alcohol, and for a drawing of the apparatus devised by him for estimating original gravities.

I trust that the object with which the book has been written, will be borne in mind by those who may give an opinion of its value, and hope that it may be found worthy of approval as well as useful.

BENJ. H. PAUL.

London, May 1857.

PREFACE BY DR. BOLLEY.

SINCE the publication of the German edition of this Manual, the labours of chemists have led to a knowledge of much that falls within the plan of the work.

The necessity of making known, as quickly as possible, whatever may be discovered or suggested in connexion with the industrial arts, and also of putting it to the test of practice, is so essentially a part of technical requirements at the present time, that the introduction into the work of results, which the progress of investigation has furnished, could not with propriety be omitted before its publication in the language of a country where the most powerful minds and the most gigantic material appliances are conjointly directed to the advancement of technical knowledge.

It was with great willingness, therefore, that I complied with Dr. Paul's wish, that the text should be revised and such new matter added as will, I hope, increase the usefulness of the book.

DR. BOLLEY.

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MANUAL

OF

TECHNICAL ANALYSIS.

INTRODUCTION.

CHEMICAL analysis, to the extent at least in which it is treated of in this work, is essentially an art, inasmuch as it is the application of the recognized principles and facts of chemical science to the solution of questions of more or less frequent recurrence and practical utility. The general object of analytical operations is to ascertain the chemical nature of substances; their special object may be either to ascertain what are the elements of which a substance consists, and what their state of combination, or, these particulars being known, to learn their relative proportions.

Thus analysis is either qualitative or quantitative. It may sometimes be desirable in both instances to extend the examination to all the elements contained in a substance, however small their proportion; sometimes it will only be necessary either to ascertain the presence or absence of some one or more elements or compounds, or to estimate the proportion of some particular constituent of the substance examined.

Complete and minute analyses of the former kind belong almost exclusively to the more purely scientific portion of chemistry, those of the latter kind relate to the technical applications of chemistry, and are had recourse to for the purpose of determining certain points which have a commercial or technical importance, rather than any direct bearing upon the cultivation of science.

The chemical nature of any substance is always ascertained by subjecting it to certain treatment called the *test*, and the presence of any element or compound is indicated by the consequent production of some change, generally

through the agency of some other substance, and always under certain definite conditions. This change, which is called the *reaction*, usually consists in the formation of a compound of the substance sought, possessing marked and distinctive peculiarities. The substances used for this purpose are called *reagents*. It is, of course, essential that the criterion by which we judge of the presence or absence of any element or compound should be infallible, and if there should be any exceptions to its general applicability, the conditions upon which they depend must be understood and provided against.

When the substance to be examined contains a great number of elements in various states of combination, its behaviour with reagents, which would otherwise indicate their presence, would in many instances be so modified or disguised as to render their detection extremely difficult and tedious. In order to obviate the inconvenience which would result from such a circumstance, the elements have been arranged in several analytical groups, according to general analogies obtaining, either between the conditions under which they yield compounds of particular types, or in the behaviour of their compounds with certain reagents, and of such a nature that the application of a particular test will at once indicate either the absence of a whole group of elements, or the presence of some or all of its members. By a systematic application of such tests it is possible, not only to learn what groups the elements of any substance belong to, but likewise to separate these groups, and thereby greatly to facilitate the special recognition of the individual elements.

As regards heavy metals, their sulphurets have been found admirably well suited to this purpose; for the light metals, their carbonates; for acids, their silver, lime and baryta salts.

The elements, belonging to groups thus separated, are then subjected to further separation, and, lastly, the several elements are obtained in such an isolated state as to be recognized without any doubt.

The qualitative examination of substances for technical and commercial purposes is made, not for the purpose of ascertaining their chemical nature, for that in all ordinary instances is known beforehand, but with reference to certain

possible or probable impurities and admixtures, either accidental or fraudulent, which may lessen their market value or prove detrimental in the operations for which they are required. The nature of these impurities or admixtures may generally be anticipated, and the necessary operations of testing are therefore brought within a more limited range than is the case in the examination of substances whose chemical nature is more or less unknown, and when the probable nature of the elements or compounds present cannot always be determined beforehand even by the professional chemist.

In the general system of qualitative examination subsequently described, those elements only have been taken into account which are likely to be of interest commercially or technically. Although a rigid adherence to the entire system is not always necessary, still, to lay down any rules for deviating from, or omitting any portion of it, would be attempting a royal road to a knowledge which the analyst can only acquire by experience and careful observation of chemical phænomena.

In quantitative analysis a knowledge of the elements present in the substances to be examined is presupposed. It is here necessary to devise means of effecting a perfect separation of each element without loss, and in such a form that its proportion can be accurately estimated. This has hitherto generally been effected by successively converting the several elements into insoluble and definite compounds which may be collected and weighed. Then by means of the law of combining proportions, the quantity of the element sought may be calculated. Thus, for instance, in the estimation of silver or chlorine, the compound weighed in either case is represented by the symbolic formula

$$Ag + Cl,$$

and substituting for the symbols their numerical equivalents,

$$107{\cdot}973 + 35{\cdot}462,$$

we obtain the relative proportion of these substances in the compound weighed, and can calculate the quantity of either constituent in the weight of chloride of silver obtained, by a simple proportion. Thus if the substance sought is silver

$= x$, and the weight of chloride of silver obtained $= y$, then

$$x = \frac{\text{Ag} \times y}{\text{Ag} + \text{Cl}} = \frac{107{\cdot}973 \times y}{107{\cdot}973 + 35{\cdot}462}.$$

If $y = 1{\cdot}726$, then $x = 1{\cdot}299$.

With good manipulation this method of analysis satisfies all the requirements of precision and accuracy. It is unexceptionable for scientific research, but for many technical purposes, where the time occupied by an analysis is as much an element of its value as accuracy, it is wholly inapplicable.

However, as the law of definite proportions obtains not only in the composition of substances, but likewise for the reactions which take place between them, and as the greater number of quantitative analytical operations which come within the province of the manufacturer are of that kind to which the term assay may be very appropriately extended, their sole object being to estimate the proportion of some one ingredient of the raw material, it is not difficult to make such an application of this principle as will meet the wants of those engaged in commerce and the arts, be free from the disadvantages of the above method, and at the same time not be inferior to it in the accuracy of its results.

According to the formula for chloride of silver,

$$\text{Ag} + \text{Cl},$$

108 parts of silver require for conversion into chloride 35·5 parts of chlorine. Now by estimating the quantity of some suitable, definite compound of chlorine—such as chloride of sodium—requisite for converting into chloride, the silver x in a given weight of the substance to be assayed, the quantity of silver x may be easily ascertained, for it will bear the same proportion to the chloride of sodium consumed $= z$ as the numerical values of their symbols,

$$x : z = \text{Ag} : \text{Na Cl}.$$

And if the chloride of sodium is added to the solution of the silver in the form of a solution, a given volume of which contains a known weight of the salt, the quantity z may be readily estimated by measuring the solution used. This method of analysis, which is called the volumetrical method, consists therefore in estimating the quantity of reagent necessary to complete some particular reaction, and is the reverse of the gravimetrical method, which consists in estimating the

quantity of some definite compound produced by such a reaction. The insolubility of chloride of silver and the rapidity with which it is deposited on agitation, render the application of this method to the estimation of silver extremely appropriate, but, as will subsequently be shown, there are few reactions of a definite character that may not with some ingenuity be made available for a similar purpose.

CHAPTER I.

THE OPERATIONS CONNECTED WITH CHEMICAL INVESTIGATIONS, AND THE APPARATUS NECESSARY FOR CONDUCTING ANALYSIS.

It has always been customary among chemists to make a distinction between the means employed for the purpose of effecting chemical alteration in substances, whether it be combination, the separation of one or more of their constituents, or their conversion into other compounds, &c. The distinction is founded upon the nature of the operations, which is twofold,—the wet process, which consists in acting upon the substance in question while in solution, and the dry process, which consists in submitting it either alone, or together with certain other substances, to the influence of heat. The operations involved in the preparation of elementary substances or chemical compounds, their detection, separation and quantitative estimation, in short, general synthetical and analytical chemistry, may be conveniently classified under these two heads, and they are so much alike in all cases, that it is advisable to obviate repetition in the description of apparatus and manipulation, by giving at the outset a brief account of those which are of most general importance, and which will be necessary in carrying out the experiments treated of in this work.

Solution.

The term solution in its widest sense may be understood to denote an homogeneous liquid mixture of any substance—solid, liquid or gaseous—with a greater or less quantity of some liquid which is called the solvent.

Solution may be of two kinds, physical or chemical; the

former is a simple mixture of the substance dissolved, with the solvent, as, for instance, a solution of salt, of sulphuric acid or hydrochloric acid gas in water. Chemical solution involves an alteration of the substance dissolved and its combination in part with the solvent, for example, the solution of marble in hydrochloric acid. All chemical solvents are themselves solutions of certain substances in water, therefore chemical solution may be regarded as the formation of a soluble substance and its subsequent physical solution in that water. Thus in the above instance it is not carbonate of lime which is in solution, but chloride of calcium.

No general law of proportionality between the solvent and substance dissolved has yet been obtained for physical solutions. The solution of a substance is said to be saturated when it cannot dissolve any more. As a general rule, solution takes place more readily with the aid of heat, and as the solvent power of liquids increases with the temperature, saturation is determined by it.

As in chemical solution there is an actual combination, this of course takes place in accordance with the law of definite proportion, so much only of the substance being dissolved as may be equivalent to the quantity of reagent present. Chemical solution is facilitated by heat, because chemical action takes place more readily at somewhat elevated temperature, but the quantity of substance dissolved by a given quantity of solvent is constant and independent of temperature.

Solution is generally effected in flasks or beakers, bedded nearly to the level of the liquid in sand contained in an iron pan, and heated by a gas burner. (Fig. 1.)

Fig. 1.

Solution is always effected more readily the greater the surface of contact between the solvent and the substances to be dissolved. The latter should consequently be exposed in the state of powder to the action of solvents whenever it is practicable. For the same reason the mixture should likewise be frequently shaken.

When in the chemical solution of substances the reaction is violent or a gas is disengaged, precautions must be taken that none of the liquid is thrown out of the vessel by spirting. In quantitative experiments this is particularly important. The best means of preventing loss in this way is to introduce the substance into a rather capacious beaker, the top of which is covered with a very short-necked funnel or a thin hemispherical glass with a hole in the centre, through which the solvent is gradually added. After the solution is complete, the portions of liquid projected against the under surface of the glass can be washed off into the beaker with a dropping bottle. (Fig. 7.)

Fig. 2.

In preparing solutions from organic substances which are but partially soluble, they are macerated or kept in contact with the solvent for some time, and the liquid then strained off. The process is termed *decoction* when the mixture is boiled; *infusion*, when conducted without the continued application of an elevated temperature.

Precipitation.

This operation is the direct reverse of solution, and is very frequently adopted for the purpose of separating substances, or of collecting them in a convenient form for weighing.

Like solution it is of two kinds. Physical precipitation is effected by altering the solvent in which a substance is diffused; thus sulphate of lime is precipitated from its solution in water by the addition of alcohol, and phosphate of lime from its solution in hydrochloric acid, by the addition of ammonia, the salts named being respectively insoluble in weak alcohol and in chloride of ammonium.

Chemical precipitation, on the contrary, involves an alteration in the substance dissolved; thus from a solution of sulphate of alumina, ammonia precipitates alumina by abstracting the acid combined with it.

The substance which, in this operation, is converted into the insoluble solid form, is called the *precipitate*, that which determines its separation, the *precipitant*.

The physical characters of precipitates are extremely varied. According to their aggregation they are called crystalline, pulverulent, curdy, flocculent, gelatinous, &c., and these characters, together with colour, are under certain circumstances trustworthy means of recognition. But it is not admissible to take these facts alone as indicative of their chemical nature. This must be confirmed by subsequent observation of their behaviour with appropriate reagents.

The vessels in which precipation is best effected, are the thin glass beakers, flasks, or test tubes (figs. 1, 5, and 8), which admit of being heated without risk of breakage, for as a general rule, precipitates separate more readily from hot liquids.

In qualitative analysis the principal points to be attended to in precipitation are the conditions under which a reagent is to be applied, for instance, the temperature, alkalinity or acidity of the solution, &c. When the quantity of a substance tested for by precipitation is very minute, it may happen that no visible precipitate is formed on the addition of the reagent, but this must not at once be taken as a negative indication; the tube must be allowed to stand some hours and then examined. If no precipitate is yet visible, the liquid should be shaken, heated, or perhaps evaporated somewhat, and again examined when cold. In some delicate experiments a neglect of these precautions will involve error.

In quantitative analysis, where it is essential that there should not be the slightest loss, either of precipitate or of the liquid from which it is thrown down, beakers are the most convenient vessels for precipitation, because, when collecting the precipitate for weighing, any particles adhering to the sides may be more easily detached by means of a feather, stripped bare with the exception of a tiny tuft at the end, and washed out by a fine jet of water from the wash bottle (fig. 6). When this is ineffectual, the adherent

particles must be dissolved off, and again precipitated. Glass rods used for stirring must always be washed before removal from the vessel containing the precipitate.

Sometimes it may be desirable to use only just so much of a precipitant as will completely precipitate the substance to be removed from solution. In such cases great caution must be exercised, and the liquid frequently cleared, either by stirring or heating, so as to show distinctly whether on the addition of more reagent any further precipitate is formed. A very delicate mode of ascertaining this is to dip into the perfectly clear liquid a glass rod moistened at one end with the precipitant.

The separation of precipitates from the liquids with which they are mixed is effected by one or other of the following operations.

Decantation.

When the precipitate to be separated is of considerably greater density than the liquid with which it is mixed, decantation is convenient, because it occupies little time. The vessels in which precipitates are to be collected or washed by decantation should have an area that is very small in proportion to their height. The clear supernatant liquid may be poured off by gently inclining the vessel, or it may be drawn off by means of a pipette or siphon. The vessel is then filled with water, the precipitate stirred with a glass rod, allowed to settle, and the water drawn off as before.

Precipitates which, like chloride of silver, do not adhere to the sides of the vessel in which they are thrown down, may be advantageously washed by decantation, and collected without using a filter, in the following manner. The precipitate is thrown down in a very narrow-necked taper flask, with an even rim capable of being closed by a watch-glass. After the washing is completed, the flask is filled with water; the mouth covered with a crucible, the whole rapidly inverted and allowed to stand until the precipitate has completely subsided into the crucible. The flask is then very carefully raised, so as to admit of a watch-glass being slipped under its mouth without disturbing the precipitate, and rapidly removed from the crucible. The water left in the crucible may be decanted off. This operation requires some

little dexterity, which, however, it is well worth while to acquire.

Filtration.

When a precipitate is deposited but slowly, recourse must be had to filtration, which is a mechanical mode of separation by means of unsized paper, and is analogous to sifting, for while the liquid passes through the pores of the paper, the precipitate is retained upon its surface.

Filtration is convenient in quantitative analysis, because the precipitate, as it is contained in a filter, can be transferred to the drying bath or crucible more readily than when collected by decantation.

The paper used for this purpose should be as much as possible free from mineral substance, thin and compact in texture, and should allow water to pass rapidly through it. It should be cut into discs from 2 to 10 inches in diameter. The filter is made by folding one of these discs diametrically at right angles, and then opening it so as to form a cone which is supported upon a glass funnel.

Fig. 3.

The funnels used for filtration should be quite even, the spout should be ground obliquely, and form an abrupt angle of about 60° with the expanded part, so that the greater part of the paper cone may fit close, while its apex remains out of contact with the funnel. The filter should never extend quite so far as the rim of the funnel.

Fig. 4.

The vessel into which a liquid is to be filtered must always be large enough to hold considerably more than the whole of it, and the spout of the funnel should rest against its side so as to prevent splashing.

In quantitative experiments any loss in transferring a precipitate to the filter must be guarded against. For this purpose the edge of the vessel should be slightly greased, and a glass rod held against it while pouring so as to direct the stream of liquid upon the thick folded side of the filter, and prevent it from piercing the paper.

Sometimes, when a precipitate is in a very fine state of division, it passes through the pores of the filter. It may happen that a double filter is the only remedy for this inconvenience, but it may often be prevented by allowing some time to elapse before transferring the precipitate to the filter, or by the application of heat. In either case the precipitate acquires a granular or crystalline condition which renders it more readily separable.

Fig. 5.

Washing Precipitates.

Precipitates, as collected upon a filter, always retain a portion of the liquid with which they were mixed, greater or less in proportion to their bulkiness. When this liquid does not contain any saline or fixed substance, simply drying the filter and its contents is all that is necessary. But as the liquid from which a precipitate is thrown down will seldom be free from dissolved substances, washing is an operation that is almost always supplementary to precipitation. It may be effected by decantation, or, when the precipitate has been collected upon a filter, by repeated addition of water, until the filtrate no longer shows indications of dissolved substances, either when evaporated on platinum foil or when tested with an appropriate reagent.

Some precipitates which are not wholly insoluble in pure water, or which are mechanically carried through the paper by it, are to be washed with a very dilute solution of some substance which is not fixed, as chloride of ammonium, nitric acid, &c.

In washing precipitates by filtration, it is generally advisable to collect them as much as possible into the corner of the filters, by means of a fine jet of water directed against the upper edge of the filter, so as to wash down the precipitate. The apparatus used for this purpose is the wash flask, which consists of a flat-bottomed flask with two tubes fitted in the neck by means of a good cork (fig. 6). One end of

the tube *abc* terminates just below the lower surface of the cork, at the other end it is bent at an angle, and the bore is contracted, so as to leave only a very small aperture at *e*. The bent tube *def* extends to the bottom of the flask. When the flask stands upon its bottom there is free communication between the exterior and interior air by the bent tube *abc*; when, on the contrary, it is reversed (fig. 7), air enters through the tube *def*, and the water flows out through the tube *abc* in a fine stream, which may be directed upon different portions of a precipitate contained in a filter, or upon the sides of a vessel, for the purpose of removing adhering particles of a precipitate. The velocity, and consequently the force of the stream, is proportionate to the difference of level *h*, and may be augmented by increasing the length of the tube *abc*.

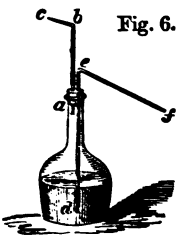
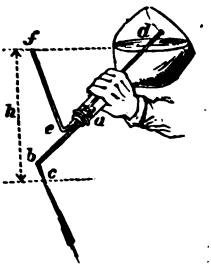


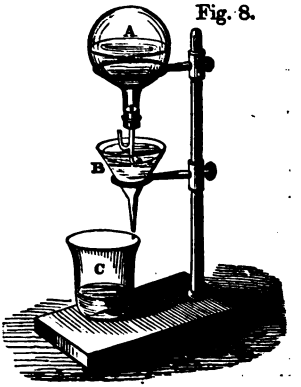
Fig. 6.

Fig. 7.



Another form of wash flask (fig. 8) is very convenient when the precipitate is gelatinous, or requires considerable washing, or when the water passes very slowly through the filter. It consists of a large flask A, into the mouth of which is fitted, by means of a cork, a tube of the form represented on a larger scale by fig. 9. The flask is completely filled with water, and inverted above the funnel in such a manner that the fine curved aperture *d* dips about half an inch below the water in the filter. It is retained in this position by means of a support. The pressure of the atmosphere is exerted equally upon the liquid in the narrow tube *bc*, and upon the surface of the liquid in the filter,

Fig. 8.



and consequently upon the liquid in the tube *abd*. The water in the flask tends to flow in virtue of the pressure of a column of water *h*, equal in height to the difference between the surface of the liquid in the filter, and the surface of that in the lateral tube *cb*; but as the tube *cb* is very narrow, the capillary action prevents air from entering the flask through it, and is equal to the pressure of a small column of water; therefore the water cannot flow from the flask so long as the capillary action in *cb* is more than equivalent to the hydrostatic pressure of the column of water *h*. In proportion as the water in the filter passes through, the height of the column *h* increases until, when its pressure just exceeds the capillary action in *cb*, air enters by the lateral tube *cb*, and water flows from the flask into the filter again, establishing the equilibrium by elevating the level of the water in the filter. When this apparatus has been adjusted, it requires no further attention, the filter is kept filled with water to an almost constant level, and the fresh water always occupies the upper portion, a circumstance very favourable for perfect washing.

Fig. 9.

Evaporation, Distillation, etc.

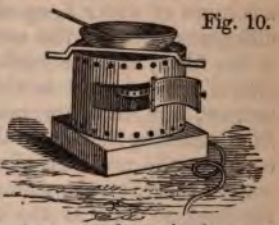
When, in the course of analysis, substances which are capable of assuming the vaporous state at a more or less elevated temperature are to be separated from others which are fixed under the same circumstances, it is often convenient to effect their separation in this manner.

When the fixed and volatile substances are in the state of physical intermixture, and it is not necessary to save the latter as in the separation of water and some dissolved substances, the operation is called *evaporation*, and is conducted in shallow porcelain or metal dishes over a gas flame. The necessity for evaporating liquids for the purpose either of concentration or of obtaining dissolved substances in a dry state, is of very frequent occurrence in analysis.

The most convenient arrangement for evaporation (fig. 10) consists of a simple cylinder of sheet iron, pierced at the top and bottom with holes, to admit a current of air, and

furnished with a circular gas burner in the middle. The whole apparatus should be placed upon a square clay slab, in order that the table may not be burned. With the aid of a triangle of iron bar and some flat rings of various sizes, it may be made to answer all the requirements of ordinary analytical operations.

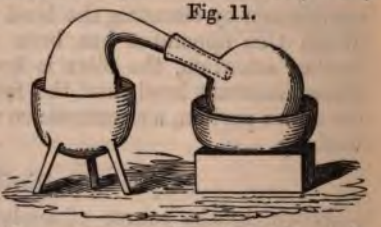
Fig. 10.



In quantitative analysis it is better to apply heat in evaporation through the medium of sand, steam, or hot air, because then the evaporation is more uniform than it is over an open fire, and there is less danger of any portion of the liquid being thrown out.

When the volatile substance is to be collected, the operation is conducted in a close vessel, either a retort, flask, or tube, and the vapour passed into another vessel, where it is condensed. The operation is termed distillation when the substance to be separated is a liquid; sublimation when it is a solid.

Fig. 11.



The general form of apparatus used for distillation upon a small scale is shown in figs. 11, 12. In many instances it is necessary to pass the

Fig. 12.

vapour through a tube cooled externally by a stream of water, in order that it may be condensed. This tube is partially enclosed by means of perforated corks within a larger metal tube DE, furnished with a spout at *f* and a funnel at *d*. This condenser is arranged in an inclined position, so that the water, heated by the condensation of vapour, rises to the higher end, and is forced out by the cold water entering at *d*. Slight modifications which may be requisite to meet particular cases, will naturally suggest themselves to the operator.

Sublimation for analytical purposes may generally be conducted in glass tubes, sealed at one end and closed at the other by the fingers.

Drying, Ignition, etc.

When in quantitative analysis, precipitates or other substances are to be weighed, it is essential not only that all adherent soluble substances should be washed out, but likewise that they should be perfectly dry.

Moreover, as most substances are more or less hygroscopic, while others contain water in a state of loose chemical combination, it is generally advisable, before weighing any substance for analysis, to ensure either the total absence of moisture, or a definite state of hydration of the substance in question. This may be effected in various ways according to circumstances.

Precipitates may be weighed in three different ways; when washed by decantation they may be transferred to a porcelain capsule or crucible of known weight in the manner above described, and after being dried by exposure to a proper temperature, weighed. When precipitates have been collected by filtration, they may be weighed, after perfect drying, together with the filters, the weight of which, in an equal state of dryness, has been previously ascertained. Results of sufficient accuracy for some purposes may be obtained by using another dried filter as a counterpoise for the one containing the precipitate; but the method more usually adopted, which involves the least probability of error, and which must necessarily be had recourse to, when the precipitate has to be exposed to an elevated temperature previous to weighing, is to burn away the paper.

Water which is merely adherent may generally be removed by exposing the substances in a confined space, to air dried by contact with a large surface of concentrated sulphuric acid, which absorbs with avidity the water suspended in the air. A very convenient arrangement for this purpose consists of a large bell glass, ground at the bottom to fit air-tight upon a glass plate, upon which stands a porcelain dish containing sulphuric acid, and furnished with a support for receiving crucibles, watch-glasses, &c. (fig. 13). A dry air-chamber of this kind is an indispensable companion to the balance, for the purpose of allowing substances, which have been dried by heat or ignited, to cool without any fear of their absorbing moisture.

Fig 13.

When water is chemically combined, and when it is in that state intermediate between chemical and mere physical mixture, as in deliquesced salts, the influence of a more or less elevated temperature and a full current of air is requisite for the perfect separation of water. For this purpose the hot air-chamber is used, and as a temperature of 212° F. is in the greater number of instances quite sufficient to ensure at least a definite state of hydration, boiling water is a very convenient medium for the application of heat. The best form of apparatus (fig. 14) consists of a square copper box, so constructed as to admit of water being boiled in a space formed by double walls on five sides of the box. The front is closed by a door with two holes to admit a current of air. In the interior, *a*, there should be a light wooden frame for holding crucibles, &c. Care should be taken that this vessel always contains water while in use, otherwise the temperature may rise far above 212° F.

Fig. 14.

When a temperature considerably above the boiling-point of water is required for drying, it is advisable to employ a simple copper box A (fig. 15) of convenient form, and heated by a gas flame. Through an aperture in the lid E, a thermometer D is introduced to indicate the degree of heat attained.

Fig. 15.

When the filter in which a precipitate is contained is to be burnt, the first step is to render it thoroughly dry by exposure in the hot air-chamber. The filter is then unfolded, and its contents transferred as perfectly as possible into a clean porcelain crucible, placed upon a sheet of highly glazed paper for the purpose of saving any minute particles which may be dropped.

The filter is again folded up into the shape of a closed fan, and ignited at the lower part, while held by the upper part with a pair of platinum pointed tongs so that the ash may fall into the crucible, which is then gradually heated to redness, and kept at that temperature until the whole of the carbon has been burnt off.

The ignition of substances for the purpose of bringing them to a definite composition requires several precautions. When water or any other constituent is separated on the application of heat, it is in the gaseous state, and if the disengagement of vapour is too rapid, portions of the substance may be projected out of the crucible, especially when the substance is in very fine powder. This danger must therefore always be provided against by first thoroughly drying the substance in an air-bath, and then raising the temperature of the crucible very slowly to the point of ignition.

In the case of substances, which, like chloride of silver, are decomposed when ignited in contact with carbon, it is desirable to avoid collecting them by filtration, but when that is impossible, they must be carefully detached from the paper, which is burnt separately upon the lid of the crucible.

Sometimes there is great difficulty in burning off the last traces of carbon from the filter ash; when long-continued ignition does not suffice for this purpose, and there is no reason to fear decomposition of the substance, the filter ash

may be moistened with nitric acid, dried, and again gently heated. As soon as the ignition of a substance to be weighed is completed, the crucible is transferred to the dry air-chamber (fig. 13) to cool.

Fig. 16.

The temperature requisite for ignition of precipitates, &c., previous to weighing, may almost always be obtained by the use of the gas furnace (fig. 16), which consists of a stout copper cylinder, 5 inches high, and 2 inches wide, fitting over a gas jet in the same way as a lamp-chimney. The bottom of the cylinder is open and admits of the mixture of air with the gas as it issues from the jet, and the top is covered with a cap of wire gauze, above which the mixture of gas and air burns. This contrivance ensures a higher temperature than is attainable with an ordinary gas-flame, and, by facilitating combustion, prevents the deposition of carbon upon the exterior of the crucibles or other vessels heated.

Crucibles may be supported in this flame upon a triangle made of three pieces of tobacco-pipe strung upon wire, the ends of which are twisted together, or when a higher temperature is required, the crucible jacket (fig. 16) may be used. This consists simply of two truncated cones of stout sheet iron, fitting together at their bases. The lower cone is furnished with a circular ledge inside, upon which the triangle rests, and a handle for removing it while hot.

Roasting.

This operation consists in exposing substances under the influence of a more or less elevated temperature, to the action of some gaseous substance, capable of combining with some of their constituents, and is adopted for the purpose either of separating them, or of converting them into some other state of combination.

The most usual gaseous reagent employed in roasting, is oxygen; the powdered substance, spread thinly over a metal or earthen plate, being heated to dull redness in the air.

The sulphur of many ores is removed in this manner previous to the assay by the dry process. It is sometimes desirable to use pure oxygen, chlorine, or some other gas, as reagent in roasting operations, and in such a case the substance is exposed to the action of the gas in a small tray of platinum or porcelain, contained in a tube of glass or porcelain, through which the gas is passed in a slow stream.

Melting.

The object of this operation may be,—1st, to convert substances under analysis into a form soluble in water or by the aid of acid; 2ndly, to effect the separation of some constituent sought to be estimated.

A great number of assays in the dry way are made by acting upon the ore in question according to the latter method. The small gas furnace (fig. 16) is capable of producing a temperature sufficiently high for some few purposes, but there are many which require such a temperature as can only be obtained in a well-constructed coke furnace.

The other apparatus requisite for fusions consists of crucibles of baked clay, iron, porcelain, or platinum. For rough operations and for metal assays the former two are best adapted, but for the more delicate operations of analysis crucibles of porcelain or platinum must be used.

Some judgement must be exercised in the selection of crucibles for certain purposes, so as to avoid, on the one hand, destruction of the crucible, and on the other, contamination of the substance under examination. Thus porcelain crucibles cannot be used for fusions in which alkalies are used or formed in the reaction Platinum crucibles must not be used for fusions in which chlorine will be generated; in which nitrate of potash, caustic alkalies, sulphurets, cyanides, salts of easily reducible metals, phosphoric or organic acids are present. So likewise, when crucibles of porcelain or platinum are to be exposed to very high temperatures in a coke furnace, it is advisable to place them within a larger earthen crucible, containing some calcined magnesia, in order to prevent the small crucible from being upset or damaged by contact with the fuel.

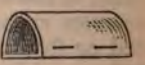
A coke furnace, made of sheet-iron, lined with clay, and of the form represented by fig. 17, will answer for most purposes in which a high temperature is required, as well as for some cases of distillation or evaporation. It should be placed upon stone slabs, near a flue, into which is inserted a piece of jointed iron pipe to carry off the smoke.

Fig. 17.

Cupellation.

This operation is a combination of roasting and melting, and relates particularly to the separation of certain metals. It is conducted in a *muffle* (fig. 18), which is a semi-cylindrical chamber of baked clay or iron fitting into the aperture M at the side of the melting furnace (fig. 17). Small shallow vessels of compressed bone-ash, called *cupels*, are used in this operation instead of crucibles.

Fig. 18.



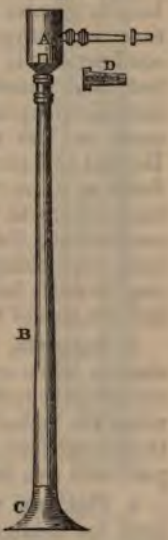
A muffle may be advantageously used for other purposes besides cupellation, as roasting, melting, ignition of precipitates, &c.

Blowpipe Operations.

These may be regarded as miniature imitations of several kinds of operations in the dry way, practised in the arts for

the extraction of metals from their ores, the production of glasses, &c. The indications which they furnish are in many instances very valuable, owing to the rapidity with which they may be obtained.

Fig. 19.



The apparatus requisite for this kind of testing consists of,—

1. **The blowpipe,** which in its simplest form is a narrow curved tube of brass, with a small orifice, through which a current of air may be forced by means of the mouth or bellows against a small flame, so as to accelerate the combustion, and increase the temperature of the flame.

The best form of mouth blowpipe (fig. 19) has a box at *a*, in which the moisture of the breath may collect, and likewise a funnel-shaped mouth-piece *c* of horn or ivory, which enables the operator to keep up a continuous and uniform blast with much less fatigue. The blast is obtained by the action of the cheek muscles, not by the lungs, air being inhaled through the nostrils meanwhile. The blowpipe should be furnished with three or four platinum nozzles *d* with various-sized orifices.

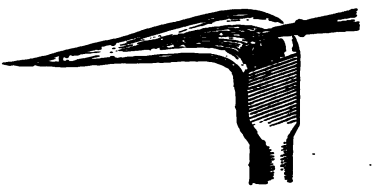
2. **The flame** to be used in blowpipe experiments may be that of a candle, oil, or gas. The latter is perhaps the most convenient, when used with a burner (fig. 20) made of a piece of tube somewhat flattened at the end, and with notches opposite each other in the direction of the greater diameter. The nozzle of the blowpipe is placed upon one of these notches, and a current of air forced through the flame, by which means it is bent down and directed against the substance to be tested.

Fig. 20.

The chemical effect produced under the influence of the blowpipe flame is of two different kinds at different parts of it, and is dependent upon the relation existing between the burning material and the current of air which supports its

combustion. In the luminous part of the flame, (fig. 21), immediately in front of the obscure cone *aa*, the carbonaceous gases are present, together with the carbon, in greater proportion than oxygen; the combustion is imperfect, and consequently metallic compounds are more or less reduced. Beyond this part of the flame at *c*, on the contrary, there is more oxygen than is sufficient for perfect combustion, the temperature is higher, and all the conditions for oxidation exist. The reducing or oxidizing flame may likewise be produced separately by regulating the blast and the size of the flame.

Fig. 21.



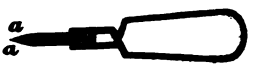
3. **Supports, &c.** Charcoal is used as a support for substances while exposed to the action of the blowpipe flame whenever reduction is to be effected, oxidation prevented, or when the reducing influence of the charcoal will not affect the experiment. It should be of fir-wood, well burnt, compact, and free from knots.

4. **Platinum wire,** about the thickness of a fine needle, is used for holding substances, while exposed, with fluxes, to influence of the flame.

5. **Platinum foil** or **spoon,** for supporting such substances as do not admit of being heated upon charcoal.

6. **Pincette.** The most convenient kind for holding small fragments (fig. 22), is so constructed, that, when at rest, the platinum points *aa* are close together, and are separated by pressure.

Fig. 22.



Measures.

Vessels of known capacity are in frequent requisition for measuring liquids. For some purposes the ordinary glass measures, used by apothecaries, answer sufficiently well. Narrow-necked flasks, with a mark upon the neck indicating a certain volume, are much more accurate. In the measure-

ment of liquids for analytical purposes, it is requisite not only that the instruments should be exact, but likewise that they should be graduated so as to indicate certain fractional parts of the volume unit.

Fig. 23.

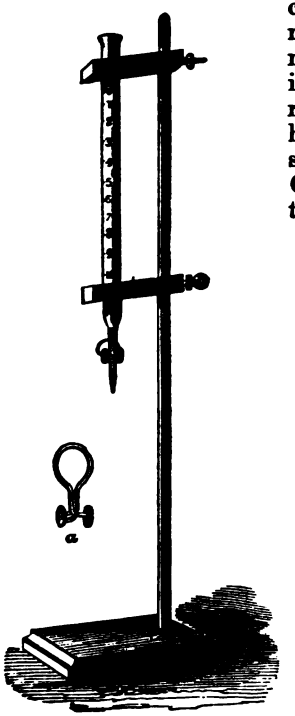
The instrument which has hitherto been most generally used for measuring liquids in analytical operations, is the *burette* of Gay-Lussac (fig 23). It is a cylindrical glass tube AB with a long spout CD attached to the lower extremity, terminating above in a curve and slightly contracted at the mouth. The burette is graduated decimally, according to any desired metrical system, and from above downwards, so as to indicate the volume of liquid poured out.

Although it cannot be doubted that this form of burette has been of very important service in the hands of chemists, it possesses some slight defects, which render it less adapted for the use of inexperienced manipulators. As it is necessary to incline the burette when pouring, the volume of liquid cannot be read off during the flow. After the liquid has been allowed to run back, the addition of small quantities is very difficult, in consequence of the frequent adhesion of a drop in the mouth of the spout, and this is a great inconvenience towards the end of an operation, when it is desirable to read off the volume frequently so as not to spoil the experiment by adding more than is sufficient to complete the reaction.

The most valuable modification in the form of the burette has been devised by Mohr (fig. 24). It consists of a tube half or three-eighths of an inch wide, contracted at the lower extremity and graduated. A short collar of vulcanized caoutchouc is fitted at one end upon the contracted neck, and a small glass spout is inserted into the other end. By means of a wire, bent in the manner shown at *a*, and furnished with knobs, the caoutchouc collar may be opened or

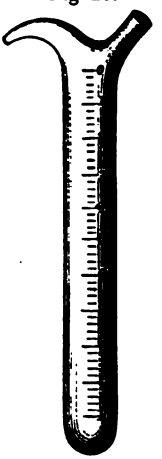
closed at pleasure so as to serve the purpose of a stopcock. The advantage of this instrument is, that very small quantities of liquid may be added from time to time, and the volume used can be observed throughout the experiment.

Fig. 24.



Unfortunately, however, it cannot be used with some volumetrical reagents, as permanganate of potash, for instance, and in such cases the form of burette represented by fig. 25 may be had recourse to, as it possesses several advantages over that of Gay-Lussac. It is less liable to fracture, and small quantities

Fig. 25.



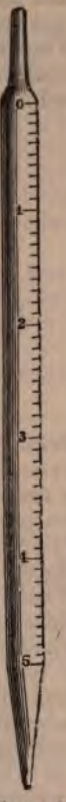
may be poured from it without difficulty.

The capacity of the burette should be from 20 to 100 cubic centimeters, and the length and width so proportioned that a small volume may be read off. The graduation should indicate 0·5 cubic centimeter, and smaller quantities may be estimated by counting the drops; a cubic centimeter contains from 12 to 16 drops. Burettes may be graduated by

measuring in successive quantities of 10 cubic centimeters, marking the spaces occupied by each with a file, and then dividing them equally into ten parts.

Fig. 26.

Fig. 27.



For measuring small quantities and for transferring liquids from one vessel to another *pipettes* are used (figs. 26 and 27). They are tubes expanded at one point into a bulb and contracted at the lower extremity or beak. They are sometimes graduated and sometimes gauged to deliver certain volumes or weights of liquid, and are filled by sucking out the air while the beak dips into the liquid. The upper orifice is then rapidly closed by the fore-finger, the liquid allowed to fall to the mark on the stem by slightly relaxing the pressure, and the measured quantity run into the vessel prepared for it.

In measuring liquids with a burette or pipette, care must be taken to have the vessel in an upright position, and that no bubbles of air remain attached to the side. The eye is then brought to a level with the surface of the liquid, and the point of the graduation, which coincides with the lowest point of the curved surface, observed.

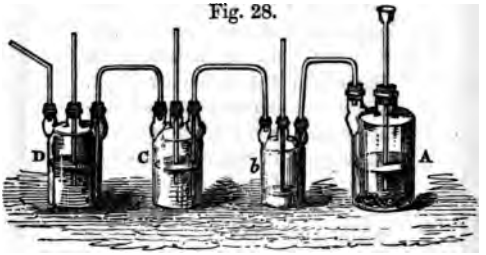
Operations with gases.

Gases are frequently used as reagents in analysis; they are generated either in vessels of the form shown at A, fig. 28,

or when the application of heat is necessary, in flasks fitted with a delivering-tube passing through a sound cork. When it is desirable to wash the gas before using it, a small three-necked bottle *b* containing a little water is arranged so that the gas has to traverse it.

When the gas is to be dissolved, as in the preparation of hydrochloric acid, ammonia, sulphuretted hydrogen, &c., it is passed into bottles CD containing water.

Fig. 28.



Weighing.

Great accuracy is requisite in the weighings made for analytical purposes; the balances used must therefore be very sensitive. For most purposes they must be capable of indicating distinctly $\frac{1}{10000}$ of the weight they are capable of carrying in each pan. For rough weighings the ordinary druggist's balance is very well adapted.

Whatever unit of weight is adopted, the weights used for chemical purposes should be decimal multiples, or submultiples of it. The French system of weights and measures is now very generally adopted by chemists, and is on every ground to be preferred.

Substances should never be weighed while hot, because by heating the surrounding air, an ascending current is produced which raises up the pan of the balance, and makes the weight of the substance appear less than it really is.

CHAPTER II.

REAGENTS AND THEIR USES.

THE reagents which are employed in analysis for the solution or chemical alteration of substances under examination, for indicating the presence of the several elements or their compounds, and for effecting their separation, must always be perfectly pure, so that the analyst may never be in any doubt as to the results which he may obtain.

The reactions which are had recourse to for the purpose of qualitative analysis are, with the exception of blowpipe tests, for the most part such as are effected by the wet process. They may be classified under the following heads:

1. Formation of an insoluble compound; precipitation.
2. Disengagement of some constituent in a gaseous state; } effervescence.
3. Alteration of colour.
4. Solution of a precipitate.

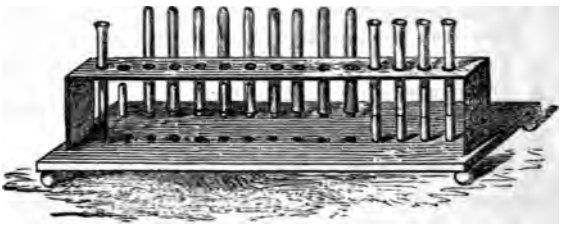
Reactions in the dry way serve less for detecting the presence of substances than as a preliminary to subsequent testing. They are for the most part essentially of the same character as the first two kinds of reactions in the wet way.

The various reactions applied in analysis take place only under certain conditions, physical and chemical, which may be more or less special; it is therefore indispensable to ensure the existence of these conditions, in testing for any substance, or in applying any chemical treatment. Reactions are likewise definite in their quantitative as well as their qualitative relations, and the analyst must acquire a capability of judging how much of a reagent will be sufficient or requisite in particular cases. The data for such an estimation, which should always be present to the mind in every analytical operation, are, the per-centage of reagent in a given volume of solution, its equivalent number, the probable or possible quantity and equivalent number of the substance sought, and lastly, the equation representing the

reaction that takes place between them. Although the addition of a larger quantity of reagent than is actually necessary may not involve any chemical inconvenience, its presence will generally become a mechanical impediment at a subsequent stage of the analysis. Moreover, such a mode of proceeding tends to confirm a slovenly and inconsiderate habit of working.

The test for any particular substance by the wet process is generally applied in a thin glass tube, closed at one end, six inches long, and from half an inch to two inches wide. These test-tubes should be arranged in a stand, capable of holding about twenty-four, either inverted on pegs, when clean and ready for use, or supported by a perforated shelf, when containing liquids under examination. (Fig. 29.)

Fig. 29.



The reagents employed in the wet process are always preserved for use in solution; those for the dry process, in the state of powder.

Water, HO. In consequence of its very general solvent property, pure water is most frequently used in chemical operations by the wet process for dissolving substances under examination, either with or without previous chemical alteration. It is likewise used in some few instances for effecting chemical alteration.

The water used for analytical operations must always be quite pure. Sometimes rain-water, after being boiled and filtered, is good enough, but it is generally necessary to purify water from its fixed and gaseous impregnations. For this purpose distillation from a copper still, with a tinned head, is the most effectual, care being taken that none of the water is thrown over mechanically into the condenser in

consequence of too violent ebullition. The first and last tenths should be rejected, and that portion only of the water collected which gives no indications of impurity when treated with suitable reagents.

Water should not leave any residue on evaporation, nor alter the colour of litmus paper. It should not become turbid on the addition of nitrate of silver, chloride of barium, or lime water.

Alcohol, ÆO, HO. This is used either anhydrous or diluted, for the separation of substances by the solution of some one or more of them, and likewise to effect the perfect precipitation of some compounds for quantitative estimation; for example, sulphate of lime, and the double chloride of platinum and potassium.

The commercial alcohol is generally pure enough for most purposes. It may be rendered anhydrous by repeated treatment with sulphate of copper which has been heated in an iron pan, until it becomes a white powder ($CuO\ SO_3\ HO$). After some days the alcohol is decanted off and distilled.

Alcohol should not redden litmus paper, nor leave any residue on evaporation.

Æther, ÆO. Used only for the separation of bromine.

The commercial æther is sufficiently pure.

Hydrochloric acid. Solution of HCl in water. This acid is very largely used as a solvent for substances which are insoluble in water. The action of hydrochloric acid in effecting solution is generally chemical and indirect, for it consists in the formation of chlorides, compounds which are characterized as a class by their ready solubility in water. The exceptions are chloride of silver and subchloride of mercury, insoluble; chloride of lead, sparingly soluble. The chlorides of antimony and bismuth, and to a less extent, the lower chloride of tin, are decomposed by water, and dissolve only in the presence of free acid.

The reaction which takes place on treating substances with hydrochloric acid varies according to their constitution. The most simple case is the solution of a protoxide:

$$1.\ MO + HCl = MCl + HO.$$

When the protoxide is combined with an acid displaceable by chlorine, it may remain in solution, or be separated in a gaseous state, or as a precipitate.

Peroxides and their salts, when treated with hydrochloric acid, give rise to the disengagement of chlorine:

$$2.\ M_2O_3 + 3HCl = 2MCl + Cl + 3HO.$$

The solution of some saline compounds by hydrochloric acid may very probably consist in the formation of an acid salt, soluble in water, together with some portion of chloride, but in the case of acids and bases which do not form soluble acid compounds, the solution must be regarded as merely physical.

Sulphuret of hydrogen, HS. This reagent is employed in qualitative analysis, either in the gaseous state or as a saturated solution in water, which takes up between two and three times its volume, for the separation of metals belonging to different groups. The reaction consists in the formation of sulphurets,

$$MCl + HS = MS + HCl,$$

and the conditions under which they are formed admit of the metals being separated with ease.

The metals which are precipitated by sulphuretted hydrogen from solutions containing a *free mineral acid* are enumerated below:—

Copper	CuS .. black.	No precipitation when a large excess of acid is present until the solution is diluted.
Lead	PbS .. „	
Bismuth	BiS_3 .. „	
Cadmium	CdS .. yellow.	
Mercury	HgS .. black.	Precipitate first white, then yellow, brown, and lastly black.
Arsenic	AsS_3, AsS_5 yellow.	In dilute solutions the precipitation is tardy: it is facilitated by heat.
Antimony	SbS_3, SbS_5 orange.	
Tin	SnS .. black. / SnS_2.. yellow.	Precipitated more readily on heating the solution.
Gold	AuS_3.. black.	
Platinum	PtS_2 .. „	Precipitation requires heat.

Sulphuret of Ammonium. Solution of NH_4S, HS in water. This reagent is employed for the separation of those metals which are not precipitated from acid solutions by sulphuret of hydrogen. They are, with two exceptions, precipitated as sulphurets, but only from solutions which are alkaline, or at least neutral, because their sulphurets are soluble in weak acids. The basic constituent of the sulphuret of ammonium serves to neutralize the acid produced in the reaction which is represented by the equation

$$MCl + NH_4S = MS + NH_4Cl.$$

Aluminium	Al_2O_3	.. white.
Chromium	Cr_2O_3	.. green.
Zinc	ZnS	.. white.
Iron	FeS	.. black.
Cobalt	CoS	.. „
Nickel	NiS	.. „
Manganese	MnS	.. flesh colour.
Uranium	UrS	.. black.

Sulphuret of ammonium is likewise employed for the further separation of the sulphurets precipitated from acid solutions by sulphuret of hydrogen, because some of them combine with it, forming soluble double compounds, while the remainder do not.

Sulphurets soluble in sulphuret of ammonium.

AuS_3, PtS_2, SbS_3 } The presence of an excess of sulphur in the reagent, indicated by a yellow colour, facilitates the solution.

SnS } Very sparingly soluble in NH_4S, except when S is present in excess, when it is converted into SnS_2.

SnS_2, AsS_3, AsS_5 } Readily soluble.

This reagent is prepared by passing washed sulphuretted hydrogen into solution of ammonia until it is no longer absorbed.

When first prepared it is colourless, and acids do not cause precipitation of sulphur. In time, however, it acquires a yellow tinge, and then acids precipitate sulphur; if this precipitate is not considerable, the reagent is fit for use.

Sulphuret of ammonium should not give any precipitate with sulphate of magnesia, nor leave any fixed residue on evaporation to dryness.

Carbonate of ammonia, NH_4O,CO_2; dissolved in 4 parts of water. This reagent is employed for the separation of barium, strontium, and calcium from magnesium (in the presence of choride of ammonium), and the alkali metals in solutions free from heavy metals.

The carbonates of barium, strontium, calcium, and magnesium are insoluble in water. The former three metals are not, however, entirely separated from acid solutions by carbonate of ammonia, without the aid of heat, owing to the formation of soluble bicarbonates. At the ordinary temperature magnesium is not precipitated at all, and only imperfectly on boiling. The precipitation of magnesium is likewise entirely prevented by the presence of ammoniacal salts, owing to the formation of soluble double compounds. Tests for purity, see Chap. IV.

Carbonate of potash, KO,CO_2, or **Carbonate of soda, NaO,CO_2**; dissolved in 6 parts of water. All the metals are precipitated as carbonates by carbonate of potash, and the colours of the precipitates are frequently distinctive. Alkaline carbonates are used as reagents in the dry way for decomposing by fusion silicates, sulphates, and other salts, &c. A mixture of equal parts of the two carbonates is most suitable for this purpose, because it fuses at a much lower temperature than either alone.

Carbonate of soda is used in blowpipe analysis as a reagent, and to facilitate the reduction of substances in charcoal.

Ammonia, NH_3, solution in water, 0·93 sp. gr. Used as a precipitant for metals, which it separates from solution in the form of oxides; some of these are soluble in excess of the reagent, and may thus be separated from others which are insoluble. The colour of the precipitate is likewise distinctive under certain circumstances.

Where it is desirable to effect the precipitation of an oxide or to neutralize an excess of acid without introducing into the liquid any fixed base, ammonia is always used.

Potash, KO, or **Soda, NaO.** Dissolved in water, they act in much the same manner as NH_3. The solubility of alumina, oxide of chromium, and oxide of lead in caustic

potash or soda furnishes a means of separating the corresponding metals and their compounds from others.

Chloride of ammonium, NH_4Cl, dissolved in 8 parts of water, is chiefly used for effecting the separation of certain oxides from others, by preventing their precipitation by some particular reagent. This is owing to the circumstance that some metals form with ammoniacal salts soluble double compounds, which are not affected by reagents in the same manner as the simple compounds of those metals. This compound is constantly formed when, in the course of analysis, hydrochloric acid is used as a solvent, and ammonia as a precipitant, or for neutralizing excess of acid, so that in some instances a further addition of the reagent is unnecessary.

Chloride of barium, BaCl, dissolved in 10 parts of water, is used for the separation of the following acids as a group from others.

$$SO_3,\quad \underbrace{PO_5,\ AsO_3,\ AsO_5,\ CO_2,\ SiO_3,\ BO_3,\ CrO_3,\ \bar{O},\ \bar{T},\ \bar{C}}.$$

Acids whose baryta salts are insoluble in water, but soluble in acids.

As a special reagent, chloride of barium and sometimes nitrate or acetate of baryta are used for the detection of sulphuric acid.

Nitrate of silver, AgO, NO_5, in 20 parts of water, is used for separating as a group,—

Cl, I, Br, Cy.

The silver compounds of these radicals are insoluble in nitric acid, and, with the exception of the iodide, soluble in ammonia. All other salts of silver, insoluble in water, are dissolved by nitric acid.

Chloride of calcium, CaCl, in 10 parts of water, is used for separating organic acids into groups, according to the conditions under which their lime salts are precipitated.

Acids whose lime salts are insoluble in water in the presence of NH_4Cl	$\bar{O}, \bar{T}$.
Acids whose lime salts are insoluble in water in the presence of alcohol	$\bar{C}, \bar{M}$.

Perchloride of iron, Fe_2Cl_3, in 10 parts of water, is used for separating succinic and benzoic acids from acetic and formic acids, in the absence of other acids.

Nitric acid, NO_5, HO, with water. This acid is employed as a solvent for sulphurets of metals of the copper group (p. 30), the only one which resists its action being sulphuret of mercury, which is separated from the others in virtue of this behaviour.

Nitric acid is always used as a solvent in the analysis of alloys (Chap. VII.), and, on account of the facility with which it yields oxygen, for various purposes of oxidation in the wet way.

For impurities and tests see Chap. III.

Sulphuric acid, SO_3, HO, diluted with 4 parts of water. The commercial acid is for most purposes sufficiently pure.

Acetic acid, Ā. This acid is useful as a solvent and for neutralizing bases, when it is desirable to avoid introducing mineral acids into the liquid.

Lime, CaO, and **Baryta, BaO,** are used in solution in testing for carbonic acid. As reagents in the dry way, they serve for the decomposition of some minerals in which the alkalies are to be estimated.

Antimoniate of potash, KO, SbO_5. Used as a test for soda, the antimoniate of which is very sparingly soluble. The reagent is best prepared as it is wanted by boiling emetic tartar with a large quantity of water, which dissolves antimoniate of potash.

Chromate of potash, KO, CrO_3, dissolved in 10 parts of water, used as a reagent for lead, the chromate of which is yellow, and insoluble in water.

Phosphate of soda, 2NaO HO, PO_5, dissolved in 10 parts of water, is used chiefly in testing for magnesium, in the presence of free ammonia, the basic phosphate of ammonia, and magnesia being insoluble in water.

Sulphate of magnesia, MgO, SO_3, used as a reagent for phosphoric acid in the presence of ammonia. *Chloride of magnesium* is sometimes preferable.

Protosulphate of iron, FeO, SO_3, used in fresh solution as a reagent for nitric acid, with which it gives an intense brown colour; and for precipitating gold.

Ferrocyanide of potassium, Cfy + 2K, dissolved in 12 parts of water. Used as a reagent for persalts of iron, with which it gives an intense blue precipitate (prussian blue).

Ferricyanide of potassium, 2Cfy + 3K, dissolved in 10 parts of water. Used as a reagent for protosalts of iron, with which alone it gives a blue colour.

Oxalate of ammonia, NH_4O, $\dot{O}$, dissolved in 24 parts of water. Used as a reagent for lime, the oxalate of which is insoluble in water.

Acetate of lead, PbO, $\bar{A}$, dissolved in 10 parts of water.

Basic acetate of lead, 3PbO, $\bar{A}$. Prepared by digesting 25 parts of litharge with 2 parts of neutral acetate of lead and 10 parts of water at a moderate heat, until the whole becomes perfectly white. The clear liquid is preserved in a well-stoppered bottle. Used as well as the former salt, for the separation of some acids, and as a reagent for sulphurets.

Chloride of mercury, HgCl, dissolved in 16 parts of water.

Bichloride of platinum, $PtCl_2$, dissolved in 10 parts of water. Used as a reagent for potassium and ammonium, the double chlorides of these elements with platinum being insoluble in weak alcohol, and of a characteristic yellow colour.

Molybdate of ammonia, NH_4O, MoO_3. Used as a reagent for phosphoric acid.

Metals: pure zinc, iron, copper, and tin in bars and clippings. Mercury likewise in a pure state.

Chlorine is prepared, when wanted, by heating together peroxide of manganese and hydrochloric acid in a flask.

Iodine, dissolved in alcohol, is used as a test for starch.

Indigo, dissolved in sulphuric acid, is used as a test for nitric acid.

Litmus, an aqueous infusion. Used for indicating complete neutralization in the volumetrical valuation of acids and alkalies. Paper, saturated with blue or red infusion of litmus, is used for ascertaining their presence in solutions.

Turmeric is likewise used as infusion and paper, for ascertaining the presence of alkalies.

Reagents for volumetrical analysis. The reactions which are available for the quantitative estimation of substances according to this method are either:

1. *Saturation*;
2. *Precipitation*;
3. *Oxidation*, or *reduction*.

The reagents employed for the purpose are solutions containing in a given volume known quantities of an appropriate reagent, capable of producing a definite reaction with the substances to be estimated.

It is consequently necessary in analysis by this method to have some means of ascertaining the precise moment at which the reaction in any particular experiment is completed. As these are always of a special nature, they will be described together with the particular processes to which they are applied.

Besides some trustworthy means of ascertaining the completion of the reactions, there are two other things indispensable for the success of experiments of this kind; viz. 1. that the value or "*titre*" of the normal solutions should be accurately known; 2. that the graduation of the burettes used should be uniform.

By means of the equation representing the reaction in any particular instance, the quantity of reagent, r', equivalent to a hundred parts of the compound c sought, may be ascertained by the proportion

$$c : r = 100 : r'.$$

Then, as the quantity of reagent in a given volume of the normal solution is known, the quantity of substance to be estimated in any experiment may be calculated from the volume of normal solution necessary to complete the reaction. If, for instance, a volume of normal solution = V is equivalent to 100 parts of a compound c, to be estimated in a substance consisting only partially of c, and it is found that the volume of normal solution requisite to complete the reaction with 100 parts of that substance is only $\frac{9}{10}$ of V, it follows that the substance contains only 90 per cent. of c.

These normal solutions are prepared by dissolving a certain weight of the *pure* reagent in a small quantity of water,

and then adding water to make up a given volume. The best way of doing this is to have a flask with a mark upon its neck (fig. 28), indicating the volume required. When the reagent is liquid and of definite composition, it may be measured in a smaller flask of the same kind.

The decimal system of weights and measures is particularly convenient for this method of analysis.

Fig. 30.

It is advisable, whenever there are no particular reasons to the contrary, to prepare the normal solution of reagent in such a way that 100 cubic centimeters are sufficient to complete the reaction with such a quantity of the substance to which it relates, as will correspond with its equivalent number, or when the normal solution is applicable to the valuation of several substances, such quantities as correspond with their respective equivalents.

In this case, however, the quantity of material taken for valuation must be regulated according to the equivalent number of the substance sought, for as 100 cubic centimeters of normal solution contain a quantity of reagent corresponding with its equivalent number, 1 cubic centimeter will represent $\frac{1}{100}$ of an equivalent of the substance sought, and thus the per-centage value of the material examined is at once shown by the volume of normal solution expressed in cubic centimeters, required to complete a particular reaction with such a quantity of it as corresponds with the equivalent number of the substance sought.

Abundant illustrations of this principle will be given in the subsequent chapters.

CHAPTER III.

ACIDS EMPLOYED IN THE ARTS; THEIR CHARACTERS, THE TESTS OF THEIR PURITY, AND ADULTERATIONS.

Sulphuric acid, SO_3. 40·0.

The ordinary sulphuric acid of commerce should be perfectly colourless, clear, and without odour; its density varies from 1·848 to 1·830.

Sulphuric acid whose density is 1·848 is a monohydrate, whose composition is

SO_3.....	40 ..	81·64
HO.....	9 ..	18·36
	49 ..	100·00

The probable impurities are,—

Nitric acid, or *nitrous acid*. They are both indicated on the addition of a concentrated solution of protosulphate of iron, by the production of a red colour.

Hydrochloric acid, less frequently present, is indicated on the addition of a drop of nitrate of silver, by a white precipitate (Ag Cl) soluble in ammonia.

Sulphate of lead is generally present in sulphuric acid made by the combustion of sulphur. It separates as a white precipitate when the acid is diluted with water, and is further indicated, when the dilute acid is treated with sulphuret of hydrogen, by a brown precipitate (Pb S).

Sulphates of potash, *soda*, or *lime*, and *other fixed substances*, which are sometimes added for the purpose of giving weak acid a factitious density, are indicated by a residue, left on evaporation to dryness upon platinum.

Sulphurous acid is indicated by the evolution of sulphuret of hydrogen, when a fragment of metallic zinc is immersed in the acid. The gas blackens a strip of lead paper held over the mouth of the test tube.

Arsenious acid is not unfrequently present in sulphuric acid, and would in many instances be highly prejudicial. Tested for in the manner described in Chapter VII.

Organic matter communicates to the acid a dark brown or black colour, but, unless the quantity is large, the acid is equally applicable for many purposes.

Fuming sulphuric acid (Nordhausen) has a density of 1·86 or 1·92. It is brownish yellow or brown, owing to the presence of a little organic substance. When diluted with water, the odour of *sulphurous acid* is generally perceptible, and the production of a reddish colour indicates *selenium*. Admixtures of *saline* or *earthy substances* are however far more important than these. They are indicated by a fixed residue left when a small quantity of the acid is evaporated to dryness in a platinum capsule. The other impurities are the same as in ordinary sulphuric acid.

Free sulphuric acid, and all soluble sulphates, even when very largely diluted, give with solution of a baryta salt—chloride, acetate or nitrate—a white precipitate (BaO, SO_3) insoluble in all acids. Sulphuric acid in solution is therefore always tested for by a baryta salt in the presence of free acid. Its separation from other acids and its estimation are likewise effected by means of this compound.

Hydrochloric acid, HCl. 36·5.

The saturated aqueous solution of the gas has a density of 1·21. It should be perfectly colourless and clear. When the density of the solution is 1·16, its per-centage composition is about

HCl.....	34
Water.....	66
	100

The probable impurities are,—

Iron, indicated by the yellow colour of the liquid and by the production of a blue precipitate (prussian blue) when the diluted acid is mixed with ferrocyanide of potassium. The ordinary acid of commerce always contains iron, but for many purposes its presence is not detrimental.

Sulphuric acid, indicated on the addition of chloride of barium, by a white precipitate (BaO, SO_3).

Sulphurous acid, indicated by the evolution of sulphuret of hydrogen when a piece of zinc is immersed in the acid.

Arsenic or *lead*, indicated on the addition of sulphuret of hydrogen solution, by a yellow precipitate ($As S_3$) or a

brown precipitate (Pb S). Marsh's test should be applied for arsenic.

Organic matter. The yellow colour of hydrochloric acid is sometimes owing to the presence of organic matter; it is indicated when the acid is evaporated to dryness in a porcelain capsule, by a slight residue which blackens when heated.

Chlorine, in a free state, indicated, when the quantity is considerable, by the smell and by the solution of gold-leaf, or decoloration of indigo solution, still better by the production of a blue colour when the dilute acid is mixed with a few drops of iodide of potassium and starch solution.

Bromine, *Iodine*, } see Chapter VIII.

Fixed substances, chloride of sodium, &c., are indicated by a residue on evaporation to dryness.

Hydrochloric acid and soluble chlorides, even largely diluted, give with soluble salts of silver a white precipitate (Ag Cl) which is insoluble in nitric acid, becomes bluish gray when exposed to sunlight, and when freshly precipitated dissolves in ammonia, consequently chlorine is tested for and separated from other substances by precipitation with nitrate of silver in the presence of free nitric acid.

Nitric acid, NO_5. 54·0.

This acid is met with in commerce in various states of dilution. The most concentrated solution has a density of 1·52 or 1·53, and, though colourless when freshly prepared, becomes yellow under the influence of light: its per-centage composition is

NO_5.....	54	.....	61·4
HO.....	9	.....	38·6
	63		100·0

The ordinary acid of commerce has a density of 1·42.

The probable impurities are,—

Chlorine, indicated when the dilute acid is mixed with nitrate of silver, by a white precipitate (Ag Cl).

Sulphuric acid, indicated, in the dilute acid, by a precipitate with baryta salts.

Fixed substances, indicated by a residue left on evaporation to dryness.

Iodine, indicated by the production of a blue colour with starch. This test is applied by neutralizing the acid with carbonate of soda, adding a few drops of sulphuric acid, and leaving the mixture, contained in a stoppered bottle, with a piece of moistened starch paper suspended over its surface for some hours in a dark place (see Chap. VIII.).

Nitric acid rapidly decolorizes solution of indigo, particularly on the application of a gentle heat, and produces an intense brown colour with protosulphate of iron. The test to be applied for nitric acid differs however according to circumstances.

Phosphoric acid, PO_5. 71·3.

Employed in pharmacy, in the solid state and in solution. It should be colourless, transparent, and free from odour, and soluble in water and alcohol in all proportions.

The per-centage composition of the solid (*glacial*) acid is

PO_5	71·3	88·8
HO.....	9	11·2
	80·3	100·0

When heated the vapour given off should not redden litmus.

The usual impurities are,—

Nitric acid, indicated, when a solution of the acid and a few drops of sulphuric acid are heated together, by the decoloration of a drop of indigo solution.

Chlorides and *sulphates*, indicated by precipitates with nitrate of silver or chloride of barium, which are insoluble in nitric acid.

Oxalic acid, C_2O_3. $\bar{O}$. 36·0.

Colourless crystals perfectly soluble in 8 parts of water at 60° F., in an equal weight of boiling water and 4 parts of alcohol at 60° F. They should not become moist when exposed to the air.

The per-centage composition of the crystals is

$\bar{O}$ (C_2O_3).....	36	57·2
3HO	27	42·8
	63	100·0

The probable impurities are,—

Nitric acid, indicated by the decoloration of a drop of indigo solution.

Fixed substances, indicated by a residue left after ignition on platinum foil.

Other organic acids, indicated by carbonization when heated.

Lead, indicated by a brown turbidity with sulphuretted hydrogen.

Oxalic acid and soluble oxalates give with sulphate of lime solution, in the presence of free ammonia, a white precipitate ($CaO, \bar{O}$).

Tartaric acid, $C_8H_4O_{10}$. $\bar{T}$. 132·0.

Large, colourless, transparent crystals, with a very acid taste, perfectly soluble in 2 parts of water at 60° F., and much more so in alcohol.

The per-centage composition of the crystals is

$\bar{T}$	$(C_8H_4O_{10})$	132	88
2HO	.....	18	12
		150	100

The usual impurities are,—

Sulphates and *chlorides*, indicated by precipitates with nitrate of silver, or chloride of barium, which are insoluble in nitric acid.

Fixed substances, indicated by a residue left after combustion on platinum foil.

Tartaric acid and soluble tartrates give with a slight excess of lime-water a white precipitate ($CaO, \bar{T}$), which is insoluble in cold caustic soda and in acetic acid.

Citric acid, $C_{12}H_5O_{11}$. $\bar{C}i$. 165·0.

Colourless crystals with a very acid taste; they effloresce in a moderately warm atmosphere, and are soluble in 0·7 parts of water at 60° F., and less of alcohol.

The per-centage composition of the crystals is

$\bar{C}i$	$(C_{12}H_5O_{11})$....	165	82·1
4HO	.....	36	17·9
		201	100·0

The usual impurities are,—

Tartaric acid, indicated by a white precipitate with lime-water ($CaO, \bar{T}$) in the cold solution.

Fixed substances, indicated by a residue left after combustion on platinum foil.

Lead, indicated by a brown precipitate with sulphuret of hydrogen.

Sulphuric acid or *chlorides*, indicated as above.

Citric acid does not give a precipitate with lime-water at the ordinary temperature, but only when the liquid is heated to 212° F.

Acetic acid, $C_4H_3O_3 . \bar{A}$. 51·0.

The liquid acid should be perfectly colourless and clear. Its strength varies greatly, and is not uniformly indicated by the density.

The so-called glacial acetic acid is a colourless crystalline mass, and its per-centage composition is

$\bar{A}$	$(C_4H_3O_3)$	51	85·2
HO	.....	9	14·8
		60	100·0

The strongest liquid acid usually met with has a density of about 1·035, containing about 35 per cent. of water.

The usual impurities are,—

Empyreumatic oils, indicated by the odour, and sometimes by a yellow tinge in the acid; likewise by a dark-coloured residue left on evaporating to dryness the neutralized acid.

Sulphurous acid, indicated by the evolution of sulphuretted hydrogen, when mixed with pure sulphuric acid and zinc.

Nitric acid, indicated by the decoloration of indigo solution.

Fixed or *non-volatile substances*, indicated by residues left on evaporation to dryness and ignition.

Sulphuric acid or *chlorides*, indicated as in previous instances.

Acetic acid, when heated with alcohol and sulphuric acid, gives off an odour of acetic æther.

Vinegar is generally the product of a peculiar fermentation of alcoholic liquids, such as wine, beer, cyder, perry, malt wort, &c. Sometimes vinegar is made by diluting acetic acid, obtained from the distillation of wood, and deprived as far as possible of any impregnation, with water, and adding other substances for the purpose of giving it colour and flavour.

There are a great many kinds of vinegar, differing in various respects according to the source from which they have

been obtained. These differences relate principally to flavour and colour. Wine and malt vinegar are preferred for culinary purposes on account of their more agreeable flavour. The latter ought to contain nearly 5 per cent. of acetic acid.

The probable impurities and admixtures are,—

Free mineral acids. These are sometimes fraudulently added for the purpose of giving vinegar a factitious strength. Vinegar will always be found to contain small proportions of sulphates and chlorides, derived partly from the substances fermented, grapes, malt, &c., and partly from the spring or river water used in the process, so that in testing vinegar due allowance must always be made for this circumstance. Moreover, the addition of $\frac{1}{1000}$th by weight of sulphuric acid is a practice legally recognized in this country. For detecting adulteration with mineral acids the following tests may be applied:—

Sulphuric acid, said to be indicated by the permanent reddening of litmus, while the red colour produced by acetic acid disappears as the paper dries. However, *tartaric acid* would have the same effect.

Runge recommends mixing some of the suspected vinegar with a little white sugar, evaporating to dryness. When free sulphuric acid is present the residue is more or less charred.

Böttcher boils the suspected vinegar with a concentrated solution of chloride of calcium. A precipitate of sulphate of lime, after the liquid has cooled, indicates the presence of free sulphuric acid. This test has the advantage of being unaffected by the presence of the sulphates in vinegar. Garnier states that when pure vinegar is boiled with a *very little starch*, merely a drop of alcoholic solution of iodine immediately produces the blue colour of iodized starch, but that when free sulphuric acid is present the colour is not produced.

Hydrochloric acid is to be tested for in the distillate from the suspected vinegar. Its presence is indicated when the white precipitate ($AgO, \overline{A}$) formed with nitrate of silver does not dissolve completely in water.

Nitric acid detected as in acetic acid.

Tartaric acid is likewise used for adulterating vinegar. It is tested for by evaporating 200 cub. cent. of the vinegar to

dryness, treating the residue with alcohol, and adding chloride of calcium to the filtered solution. The presence of tartaric acid is then indicated by a precipitate of (CaO, $\overline{T}$).

The adulteration of vinegar with tartaric or sulphuric acid may likewise be ascertained by a volumetrical estimation of the acid which remains after the acetic acid has been separated from a known quantity of it by evaporation. Another experiment with an equal quantity of the vinegar in its natural state will give the total quantity of acid present, and from the two results the acetic acid is ascertained by the difference.

Hot flavouring substances—pepper, capsicum, &c.—may be detected by the taste of the residue left on evaporation to dryness.

The question whether a sample of vinegar has been prepared from malt, wine, beer, sugar, or other substances, may sometimes be decided by the examination of the residue left on evaporation.

Wine vinegar yields a residue containing tartar, and having a somewhat astringent taste.

Beer vinegar yields a bitter residue without tartar.

Wood vinegar yields the smallest proportion of residue, which is frequently rather empyreumatic.

Starch-sugar vinegar, and sometimes beer vinegar, give, when mixed with twice their volume of alcohol (0·832 sp. gr.), a flocculent precipitate which is very slowly deposited; it consists of dextrine, and is more bulky than the precipitates formed in the same manner in other kinds of vinegar.

CHAPTER IV.

THE ALKALIES AND THEIR COMPOUNDS; THE TESTS OF THEIR PURITY AND ADULTERATIONS.

Potassium compounds.

Carbonate of potash, KO, CO_2. 69·2. $KO, CO_2 + 2HO$. 87·2.

The pure salt should be perfectly white and wholly soluble in less than its weight of water.

The ordinary impurities are,—

Silica, remains undissolved in the form of white flocks when the salt is neutralized with hydrochloric acid, evaporated to dryness, and treated with water.

Sulphuric acid, indicated by a white precipitate (BaO, SO_3), with chloride of barium, after the addition of an excess of hydrochloric acid.

Chlorides, indicated by a white precipitate (Ag Cl), with nitrate of silver, after the addition of an excess of nitric acid.

For many purposes the presence of minute traces of sulphuric acid or chlorides is not of any consequence.

The substance known in commerce under the name of potash is a mixture of carbonate of potash with several other substances. It is never wholly soluble in water, but the quantity of insoluble substance, which is wholly valueless,—consisting of silica, carbonates, and phosphates of lime and magnesia, oxide of iron, clay, sand, and carbon,—is sometimes very small, and in good samples should not amount to more than 1 or at the utmost 3 per cent.

The soluble portion, even of potash which is not adulterated, does not consist entirely of carbonate of potash, but contains more or less chloride of potassium, sulphate and silicate of potash, and with these may be associated carbonate of soda, phosphate of potash, sulphuret of potassium, caustic potash, and organic substances.

Besides these substances, originating from the ashes or the mode of preparation, and diminishing the value of the alkali, adulteration is sometimes practised. The substances

employed for this purpose are chloride of sodium, soap-boilers' lye, soda-ash, sand, chalk, &c.

The potash of commerce sometimes contains caustic alkali. Its presence is detected by mixing a solution of one part of the alkali with a solution of two or three parts of chloride of barium; the precipitate formed, consisting for the most part of carbonate and sulphate of baryta, is separated by filtration, and when the alkali contains caustic potash a slip of turmeric paper dipped into the clear filtrate becomes brown.

Sulphuret of potassium is indicated by the disengagement of sulphuretted hydrogen when the alkali is treated with an acid.

Chloride of sodium is sometimes fraudulently mixed with potash to the extent of 70 or 80 per cent. It may be detected by neutralizing a portion of the alkali with hydrochloric acid and then adding a solution of antimoniate of potash, which gives a precipitate when soda is present. Since however unadulterated potash contains a certain per-centage of soda salts, it is necessary in making this experiment to compare the precipitate formed with that yielded by an equal quantity of potash, known to be genuine, and treated in the same way.

Sulphate of soda is detected in the same manner as the chloride. The alkalimetric valuation, p. 57, would indicate the inferiority of alkali adulterated with either of these salts, but when the adulterating substance is soda-ash, the alkalimetric result gives no indication of its presence. When therefore a sample of potash gives more than a normal precipitate with the antimoniate, and does not otherwise appear to be inferior in value, there is reason to suspect adulteration with soda-ash, and a special experiment must be made to decide this point (p. 62).

Nitrates of potash or *soda* may be detected by adding a slight excess of sulphuric acid and some solution of protosulphate of iron, which gives a deep red colour when nitric acid is present.

Bicarbonate of potash, $\left\{\begin{matrix}HO,CO_2\\KO,CO_2\end{matrix}\right\}$. **100·2.**

Should be white and perfectly soluble in 4 parts of water. It should not deliquesce in a moist atmosphere, and its solution treated with bichloride of mercury should not give a

precipitate, but at the most only become turbid (showing the absence of carbonate). Sulphuric acid and chlorine are tested for as in the carbonate.

Caustic potash, KO, HO. 56·2.

Whether solid or in solution, should not effervesce with acids (absence of carbonate) or evolve sulphuretted hydrogen. The solid potash should dissolve completely in water or alcohol, without leaving any yellow flocks (peroxide of iron).

Sulphate of potash, KO, SO_3. 87·2.

Should be colourless, not redden litmus paper, not give any precipitate with sulphuret of ammonium (metallic salts), with carbonate of soda (alkaline earths), with nitrate of silver (chlorides), or with antimoniate of potash (soda salts). With the exception of an acid reaction, the bisulphate should possess the same characters.

Chlorate of potash, KO, ClO_5. 122·7.

White scaly crystals which should not deliquesce; the solution should not give any precipitate with nitrate of silver (chlorine). After ignition of the salt its solution should not redden turmeric paper (indicating the presence of nitrate of potash, which is converted by ignition into caustic potash), become turbid with sulphuretted hydrogen (copper or lead), nor give any precipitate with chloride of barium (sulphates).

Nitrate of potash, KO, NO_5. 101·2.

Should dissolve in water without leaving any residue, should not deliquesce, nor give any precipitate with nitrate of silver (chlorides), chloride of barium (sulphates), or carbonate of soda (lime or magnesia).

Iodide of potassium, KI. 166·2.

Should be white, colourless, and completely soluble in water.

Carbonate of potash, indicated by an alkaline reaction and by deliquescence.

Iodate of potash, indicated by a brown coloration of the solution on the addition of hydrochloric or tartaric acid.

Sulphates, indicated by a precipitate with chloride of barium in the presence of hydrochloric acid.

Sulphur and *organic substances*, indicated by the offensive odour evolved and by a grey colour when the salt is melted.

Chlorides, indicated by the partial solubility in ammonia of the precipitate produced with nitrate of silver, and by a white precipitate on the addition of nitric acid to ammonia, with which the precipitate produced by nitrate of silver has been treated.

Cyanide of potassium, K Cy. 65·2.

Should be perfectly white and readily soluble in water. The fresh salt is without odour, but when it becomes moist it evolves a smell of hydrocyanic acid.

The probable impurities are,—

Carbonate of potash, indicated by its insolubility in weak alcohol.

Sulphuret of potassium, indicated by a dirty-brown precipitate on the addition of acetate of lead.

Sulphate of potash.

Chloride of potassium, indicated, after igniting the cyanide with twice its weight of nitrate of potash and ten times its weight of carbonate of soda, and adding an excess of nitric acid, by a white precipitate with nitrate of silver.

Ferrocyanogen, indicated by a blue precipitate on the addition of bichloride of iron and hydrochloric acid.

Sulphocyanogen, indicated, after the addition of an excess of hydrochloric acid, by the production of a red colour with persalts of iron.

Silica, remains as an insoluble residue when the solution is mixed with excess of hydrochloric acid, evaporated to dryness, and again treated with water.

Cyanate of potash, indicated by effervescence when the solution of cycanide in alcohol of 0·84 sp. gr. is mixed with hydrochloric acid.

Formiate of soda, indicated by the blackening of the cyanide when heated to redness.

Oxalate of potash, $KO\,\bar{O} + aq.$ 92·2.

The solution should not redden litmus.

Binoxalate of potash, $\left\{\begin{matrix} HO, \bar{O} \\ KO, \bar{O} \end{matrix}\right\} + 2\ aq.$ 146·2.

The probable impurities are,—

Bitartrate of potash, indicated by the blackening of the salt when heated.

Quadroxalate of potash is detected, when a portion of the salt is ignited, dissolved in water, and mixed with an equal quantity of the original salt, by the acid reaction of the mixture.

Quadroxalate of potash, $\left\{ \begin{matrix} 3\,HO, \bar{O} \\ KO, \bar{O} \end{matrix} \right\}$ **+ 4 aq. 254·2.**

This is the salt usually met with in commerce.

Tartrate of potash, $(KO)_2\,\bar{T}$**. 226·4.**

Should be white, soluble in its own weight of cold water and without acid reaction.

Bitartrate of potash, KO, HO, $\bar{T}$**. 188·2.**

The pure salt should be perfectly white.

The usual impurities are,—

Tartrate of lime, indicated by a precipitate with oxalate of ammonia.

Chlorides and *sulphates*, indicated in the presence of free nitric acid by precipitates with AgO, NO_5 and BaCl.

Metals, indicated by a precipitate with HS.

The crude tartar of commerce should leave after ignition a residue containing carbonate of potash, amounting to 30 per cent. of the original substance.

Potash salts in solution are recognizable by their not giving a precipitate with carbonate of soda or ammonia, and by the formation of a yellow precipitate with chloride of platinum mixed with alcohol.

Sodium Compounds.

Carbonate of soda, NaO, CO_2**. 53·0.** NaO, CO_2 **+ 10 aq. 143·0.**

The pure salt should be perfectly white, soluble in 2 parts of cold water. The soda salt of commerce should be nearly anhydrous; the crystallized salt contains about 63 per cent. of water.

The usual impurities are,—

Chloride of sodium, indicated by a white precipitate on the addition of nitrate of silver to the solution mixed with excess of nitric acid.

Sulphate of soda, indicated by a precipitate with chloride of barium in the presence of free hydrochloric acid.

Hyposulphite of soda, indicated by evolution of sulphurous acid, and after some time deposition of sulphur on the addition of hydrochloric acid.

Ferrocyanide of sodium, indicated by a blue or green colour on the addition of a mixture of proto- and persalt of iron and hydrochloric acid.

Potash salts, indicated by the precipitate formed on adding an excess of tartaric acid and alcohol.

Carbonate of lime or *magnesia* may be present, as they are slightly soluble in carbonate of soda. Oxalic acid indicates the presence of lime by a precipitate.

Caustic soda is detected in the same manner as caustic potash in potashes.

The soda of commerce contains most of these impurities, but the salt used in pharmacy and as a reagent should be quite free from any of them; so likewise

Bicarbonate of soda, $\left\{\begin{matrix}NaO,CO_2\\HO,CO_2\end{matrix}\right\}$. **84·0.**

Should not contain any of the above impurities, nor any carbonate or sesquicarbonate of soda; it should not therefore make turmeric paper brown, nor give a precipitate with a dilute solution of chloride of mercury; with a stronger solution of chloride it gives a precipitate which is at first white and gradually becomes reddish-brown, while carbonate or sesquicarbonate immediately produce a reddish-brown precipitate.

Caustic soda, NaO, HO. 40·0.

Now occurs frequently in commerce. Treated with acids it should neither effervesce (carbonic acid), nor disengage an odour of sulphuretted hydrogen (sulphuret of sodium) or sulphurous acid (hyposulphite of soda). Sulphates and chlorides are detected as in soda.

The valuation of soda alkalies is effected in the same manner as that of potash alkalies (p. 55).

Biborate of soda, $NaO,2BO_3$ + 10 aq. 190·8.

Should be quite white and soluble in 20 parts of water. The residue left after ignition should amount to 52 per cent.

The probable impurities are,—

Nitrate of potash, indicated by deflagration when the salt is thrown upon ignited coals.

Alkaline earths, indicated by a precipitate with carbonate of soda.

Sulphates and *chlorides*, indicated by precipitates with chloride of barium and nitrate of silver in the presence of free acid.

Alum, indicated by the astringent taste and acid reaction upon litmus.

Phosphate of soda, indicated by a yellow granular precipitate on the addition of molybdate of ammonia to the solution mixed with excess of nitric acid. This impurity sometimes amounts to as much as 20 per cent.

The method of valuation is described at p. 86.

Phosphate of soda, $(NaO)_2 HO, PO_5 + 24$ aq. 358·3.

Should be colourless, perfectly soluble in 4 parts of water at 60° F. The solution should not effervesce with acids, nor turn turmeric paper brown. The usual impurities are,—

Sulphates and *chlorides*, indicated by precipitates with chloride of barium in the presence of free hydrochloric acid, or with nitrate of silver in the presence of nitric acid.

An impure phosphate of soda occurs in commerce containing lime salts, chlorides, and sulphates; but it should be free from carbonate of soda, and consequently not effervesce with acids.

Sulphate of soda, $NaO, SO_3 + 10$ aq. 161·0.

The neutral salt should be in colourless crystals, perfectly soluble in 2 parts of water and without acid reaction.

The usual impurities are,—

Sulphate of magnesia, indicated by a precipitate with carbonate of soda.

Metallic salts, indicated by a precipitate with sulphuretted hydrogen.

Ammoniacal salts, indicated by the evolution of ammonia when treated with caustic potash.

Chlorides, tested for as above.

Hyposulphite of soda, NaO, S_2O_2. 79·0.

Should be colourless, unalterable when exposed to the air, readily soluble in water, insoluble in alcohol, and without alkaline reaction.

The usual impurities are,—

Sulphite of soda, indicated by deliquescence of the salt.

The solution of this salt should dissolve iodine without becoming coloured and without depositing sulphur (indicative of *sulphate of soda* or *sulphuret of sodium*).

Sulphuretted hydrogen, indicated by the immediate brown colour of the precipitate with acetate of lead.

Nitrate of soda, NaO,NO_5. 85·0.

Should be colourless, transparent, readily and perfectly soluble in water; the solution should be neutral.

The usual impurities are,—

Chloride of sodium, indicated by a precipitate with nitrate of silver. The commercial salt is seldom free from chloride of sodium, but it should not amount to more than 2 per cent.

Sulphates, indicated by a precipitate with chloride of barium.

Iodine, tested for in the same manner as in nitrate of potash.

Chloride of sodium, Na Cl. 58·5.

Should be white, unaltered by exposure to the air, and perfectly soluble in water.

The usual impurities are,—

Sulphates, indicated by a precipitate with chloride of barium.

Chlorides of calcium and *magnesium*, indicated by a precipitate with carbonate of soda.

Iron, indicated by a blue precipitate with ferrocyanide of potassium.

Copper, &c., indicated by a precipitate with sulphuretted hydrogen.

Iodine, tested for as in nitrate of potash.

The quantity of sulphate of lime may be estimated by treating a known weight of the chloride with a saturated solution of sulphate of lime, collecting the residue on a filter, where it is washed with the sulphate solution, drying and weighing.

Solutions of soda salts do not give any precipitate with carbonate of ammonia, nor with chloride of platinum and alcohol, as potash salts do; with antimoniate of potash on the contrary, when not too dilute, they give a white precipitate.

Ammoniacal compounds.

Ammonia, NH_3, 17·0, or in solution **NH_4O. 26·0.**

The solution should be a clear colourless liquid without any empyreumatic odour, and volatilizable without any residue.

The usual impurities are,—

Carbonate of ammonia, indicated by a precipitate with lime-water.

Lime, indicated by a precipitate with oxalate of ammonia after neutralization with nitric acid.

Chlorides and *sulphates*, tested for in the usual manner.

Metallic impregnations are indicated by a precipitate with sulphuretted hydrogen. It sometimes contains traces of lead.

The solution of ammonia of commerce is rarely free from some one or all of these impurities. Their presence in minute quantity does not lessen its value for most technical purposes, but when used in analysis it is indispensable that it should be absolutely pure.

Carbonate of ammonia, $2NH_4O, 3CO_2$. 188·0.

Should be perfectly colourless, translucent, readily soluble in water, and have a strong ammoniacal odour. The salt is very frequently covered with a white powder, which colours turmeric less deeply and has a less pungent odour.

The usual impurities are,—

Chlorine,
Fixed substances, especially metallic impregnations. } They are tested for as in the case of ammonia.

Chloride of ammonium, NH_4Cl. 53·5.

Should be colourless, translucent, and inodorous, not deliquescent, nor alkaline to litmus; readily soluble in hot water, and volatilizable without residue.

Impurities and tests are the same as in the other ammoniacal compounds.

Ammoniacal salts give with caustic alkalies, even at the ordinary temperature, pungent vapours of ammonia, which assume the appearance of a white cloud when a rod moistened with hydrochloric acid is brought near. Solutions of these salts give a yellow precipitate with chloride of platinum and alcohol, from which, when dried and heated, evolves a white vapour of chloride of ammonium.

CHAPTER V.

THE VALUATION OF ALKALIES, ACIDS, AND THEIR COMPOUNDS.

Alkalimetry.

THE process of alkalimetry is based upon the facts, that the compounds of the alkalies with acids are constant and definite in their composition, and that their carbonates are readily decomposed by strong acids and converted into neutral compounds. The change may be generally represented by the equation

$$MO, CO_2 + \text{acid} = MO\ \text{acid} + CO_2.$$

This reaction may be taken advantage of for the analysis of substances whose value depends upon the proportion of caustic or carbonated alkali which they contain, in two ways, viz. by estimating either,—

1. The quantity of acid requisite for neutralizing the alkali in a given quantity of the substance; or

2. The quantity of carbonic acid disengaged from a given quantity of the alkali by an excess of some stronger acid.

The latter method is applicable only when the alkali is in the state of carbonate, and the estimation of the carbonic acid must be made by weighing; but the former, or

Volumetrical method, is applicable to the valuation of either carbonated or caustic alkalies, and the marked effects of very minute quantities of acid or alkali upon the colouring principle of litmus furnish a very delicate means of ascertaining the point of saturation. Sulphuric acid is generally used for the purpose, and the normal solution is prepared according to the principle laid down in the chapter on reagents.

A given quantity of any acid requires for neutralization a quantity of any caustic or carbonated alkali, which bears the same proportion to it as the equivalent number of the alkali bears to that of the acid. In the case of sulphuric acid,

49 parts by weight will be neutralized by the following quantities of the alkalies respectively:

grms.		grms.	
49 SO_3HO containing 40 SO_3	equivalent to	47·20	KO.
		69·20	KO,CO_2.
		91·20	$KO\,HO,2CO_2$.
		31·00	NaO.
		53·00	NaO,CO_2.
		84·00	$NaO\,HO,2CO_2$.
		17·00	NH_3.
		26·00	NH_4O.
		188·00	$2NH_4O,3CO_2$.

The normal solution for alkalimetry is therefore prepared by diluting 49·00 grms. of pure sulphuric acid (sp. gr. 1·848) with water to the volume of one litre.—100 cubic centimeters of this solution will be equivalent to $\frac{1}{10}$ of the above quantities, and each cubic centimeter equivalent to $\frac{1}{1000}$ of the same.

Fig. 31.

The acid may be measured in a narrow-necked flask (fig. 31) gauged for the purpose; mixed with rather less than the necessary quantity of water, and when the temperature has fallen to 60° F., the volume made up to one litre.

The normal solution should be kept in a well-closed vessel, and before being used its value or "titre" should be verified by a direct experiment with a known quantity of pure alkali.

The amount of any alkali x may then be estimated by multiplying the quantity of alkali to which one cubic centimeter is equivalent, by the number of cubic centimeters C_n required for neutralization; the general formula being $x = a \times C_n$. Thus, in the case of a solution containing potash, if 28·3 cubic centimeters of normal solution were required, x (KO) would be 1·335 grm.,

$$x = 0{\cdot}0472 \times 28{\cdot}3.$$

When a more minute estimation is desirable, two experiments should be made, one in the ordinary way, which gives a tolerably approximative result; then, in the second experiment, a volume of normal solution, rather less than has been

found sufficient for neutralization in the former experiment, is measured by a pipette, as described under the estimation of silver (Chap. VII.), and added to the solution, the neutralization being completed with a solution only $\frac{1}{10}$ the value of the normal solution. Thus, in the above instance, 25 cubic centimeters of normal solution would be added in the second experiment, and, supposing the first result exact, 33 cubic centimeters of decimal solution would be requisite to complete the neutralization.

If the alkaline solutions in each experiment were accurately-measured fractions of a solution obtained in the course of some analysis or operation, in which it was desirable to ascertain the amount of alkali, this is of course to be found by multiplying the alkalimetric result by a number corresponding with the fraction.

The application of this method will be shown in a subsequent chapter.

When the per-centage of alkali x' is sought, the quantity of substance q operated upon must be known, and it is then calculated by a simple proportion from the quantity of alkali found $= x$, for

$$x' : 100 = x : q;$$

or it may be ascertained directly from the number of cubic centimeters of normal solution required for neutralization when q represents the equivalent of the alkali sought.

Quantity of Material. The numbers above therefore indicate for each of the alkalies the quantities q that must be taken by weight of substances in which the amount of any one of them is to be estimated, when it is desired that the number of cubic centimeters of normal solution required for neutralizing shall give at once the per-centage of alkali sought.

Thus, for instance, in the valuation of commercial potash it may be required to ascertain,—

1. The per-centage of *caustic alkali.*
2. The per-centage of *carbonated alkali.*

In the former case 4·72 grms. and in the latter 6·92 grms. of the raw material are to be taken, for as 100 cubic centimeters of normal solution neutralize exactly these quantities of the pure alkalies, each cubic centimeter will represent 0·0472 KO, or 0·0692 KO, CO_2.

If, then, in any experiment the 6·92 grms. require for neutralization 58·6 cubic centimeters of normal solution, the substance would contain 58·6 per cent. carbonate of potash, for

0·0692 × 58·6 = 4·11512, and 4·11512 : 6·92 = 58·6 : 100.

To ascertain the per-centage of potash 4·72 grms. are to be taken; an alkali with 58·6 per cent. of carbonate would require 40 cubic centimeters for neutralization, indicating 40 per cent. KO, for

0·0472 × 40 = 1·888, and 1·888 : 4·72 = 40 : 100.

When the substance under examination contains but a small amount of alkali, it is always advisable to take a multiple of the above quantities twice, ten, or twenty times as much, according to circumstances. This quantity is treated with hot water, the insoluble residue separated and washed by filtration, and the whole or a part of the clear solution used for the experiment. The per-centage result is then obtained by dividing the number of cubic centimeters of normal solution required by the multiple used in any particular case. If, for instance, the solution from 69·2 grms. of an ash required for neutralization 100 cubic centimeters of normal acid, it would contain 10 per cent. of carbonate of potash.

Sometimes it is necessary to repeat the experiments made for the valuation of alkalies. When this is probable, it is convenient to weigh two or four times as much of the substance as is needful for one experiment, to dissolve it in water, separate any insoluble residue by filtration, and then to take such a fraction of the solution as will represent the weight of substance to be operated upon. Thus, in valuation of commercial potashes, 27·68 grms. may be treated in this way, and one fourth of the solution used for an experiment.

Caustic lye must be taken in multiple quantities according to the strength, which is approximatively indicated by the density.

Density.	Quantity for experiment.
Under 1·08.....	50 cubic centimeters.
1·08 to 1·2.....	20 to 25 „ „
1·2 to 1·3.....	18 to 20 „ „

In this way the per-centage of caustic alkali for a certain

volume of lye is ascertained, and this is generally the most convenient form of expression for the result.

In the valuation of ammoniacal solution likewise, multiple quantities must be taken, since even the most saturated solution of ammonia (NH_3) does not contain more than 32 per cent. by weight of the dry gas. When the solution to be valued is strong, it should always be diluted with three or four times its volume of water, otherwise ammonia would be lost.

Fig. 32.

The experiment is conducted as follows. The solution of alkali is introduced into a flask or beaker of such capacity that there will be no risk of loss from effervescence. A similar vessel containing an equal volume of water is placed beside it upon a sheet of white paper, a few drops of tincture of litmus added to each liquid, and red colour given to the litmus in the water by a single drop of normal acid. The burette (fig. 32) is then filled to the 0 mark with normal acid, which is gradually added to the solution of alkali, towards the end of the experiment, drop by drop.

The appearances which present themselves, and to which especial attention must be directed, are the following:—

In the valuation of carbonated alkalies, the first addition of acid produces but a slight effervescence in consequence of the formation of bicarbonate of the alkali, but a point is soon reached at which the effervescence becomes brisker, and the litmus begins to assume a claret tint; about one half of the alkali will then be neutralized, while the remainder will be in the state of bicarbonate. The volume of normal

solution used is then to be observed; it will amount to about $\frac{1}{10}$ of the quantity requisite for perfect neutralization. The further addition of acid causes a brisk disengagement of carbonic acid, which gives the claret colour to the litmus. When the effervescence at the spot where the acid falls begins to be less distinct, not more than a drop or two must be added at a time; after each addition the liquid should be agitated by giving the flask a rotary motion, and the progress of the neutralization observed by drawing the end of a glass rod that has been dipped into the liquid along the surface of litmus paper. When the claret colour of the liquid suddenly passes into red, the paper is permanently reddened, the neutralization is complete, and the volume of normal solution used is read off and noted. A deduction of two drops, $\frac{1}{8}$ cubic centimeter, is then to be made for each of the streaks upon the litmus paper that remain red after drying, and another two drops as a correction for the retarding influence which sulphates exercise upon the reddening of litmus by acids.

In the case of caustic alkalies, the claret colour is not produced, and there is a sudden transition from blue to red. In other respects the experiment is the same for all alkalies.

Estimation of Water. When it is required to ascertain the per-centage of water in a sample of alkali, a weighed quantity is heated in a porcelain capsule; the loss of weight gives the amount of water.

Sources of error and collateral operations for the correction of alkalimetric results in certain cases.

Both the raw potash and soda of commerce sometimes contain substances which behave with acids exactly in the same manner as the pure alkalies. The presence of such substances would lead to a false result, inasmuch as by appropriating a portion of the normal solution, the per-centage result obtained would be too high. It is therefore necessary in all instances, before entering upon the valuation of any sample of raw alkali, to ascertain by means of some preliminary tests, whether any substances are present which would affect the result obtained by the volumetrical experiment.

The probable impurities of the several alkalies, and the

means of detecting them, have already been pointed out, and it will only be necessary here to refer to those which may influence the estimation of the alkali, and which it may be necessary to estimate quantitatively, in order to correct the error resulting from their presence.

Some of these substances are insoluble—*carbonates of lime* and *magnesia*; and they may consequently be removed by filtering the solution of alkali before adding the normal solution.

Others are soluble—*alkaline silicates* and *phosphates*; these sometimes amount to as much as 1 per cent., and no sufficiently simple means has yet been devised for obviating the error arising from their presence. In cases where it is desirable, they may be detected and estimated according to the methods described in Chap. VIII., and a corresponding correction made upon the result obtained by the volumetrical experiment.

Alkaline sulphurets are very likely to be present, especially in the raw soda of commerce, owing to a partial decomposition of the basic sulphuret of calcium, when the water used for extracting the alkali in the process of fabrication was too hot. *Alkaline sulphite*, and even *hyposulphite*, arising from the oxidation of sulphuret, may likewise be present. All these substances are decomposed by sulphuric acid in the same manner as the alkaline carbonates, but the error which they would thus cause may be prevented by igniting the alkali with a small quantity of chlorate of potash in a porcelain crucible. Alkaline sulphurets and sulphites are thus converted into sulphate, while the chlorate used remains in the state of chloride, neither salts having any influence upon the volumetrical result.

If, however, hyposulphite is present—which is fortunately seldom the case—there is formed on ignition with chlorate twice its equivalent of sulphuric acid, half of which neutralizes a part of the alkali, in consequence of which the value of the substance is correspondingly under-estimated.

When these sulphur compounds are present, it is sometimes desirable to estimate their amount, and in that case a double volumetrical experiment must be made, first with the alkali in its natural state, and then with alkali that has been ignited with chlorate. When the impurity is sulphuret

alone, the difference between the two results gives the amount of soda equivalent to it. When it is sulphite, double the difference gives the amount of soda present as sulphite. Thus, in the case of a sample of soda containing 20 per cent. of soda existing as carbonate, and 10 per cent. of soda existing as sulphuret or sulphite, the results would be,—

Before ignition.

20	p. c.	NaO	comb. with	CO_2	require	20	cubic centimeters of normal solution.
10	„	„	„	HS	„	10	„
						30	
20	„	„	„	CO_2	„	20	„
10	„	„	„	SO_2	„	50	„
						25	

After ignition.

20	p. c.	NaO	comb. with	CO_2	require	20	cubic centimeters of normal solution.
10	„	„	„	SO_3	„	none	„
						20	
20	„	„	„	CO_2	„	20	„
10	„	„	„	SO_3	„	none	„
						20	

On the gradual addition of the normal acid to soda containing sulphite only, one half of the soda of this salt is neutralized, the other half remaining more or less completely in the state of bisulphite.

Another probable admixture in potash, which would be a very fertile source of error, is *carbonate of soda*. When the presence of soda to any amount has been detected in a sample of potash, the volumetrical experiment is made as usual, and the result corrected by means of a control experiment, the object of which is to determine the per-centage of soda.

For this latter purpose a great number of methods have been proposed. One is based upon the fact that a saturated solution of sulphate of potash dissolves sulphate of soda, but not sulphate of potash. For this experiment the alkaline carbonate must be converted into sulphate. The alkalimetrical experiment having been already made, a weighed

quantity of the potash is mixed with water and a slight excess of sulphuric acid; the liquid evaporated to dryness, and the residue ignited and weighed. The powdered saline mass is then placed in a graduated cylindrical vessel, six times its weight of a saturated solution of sulphate of potash added, and the whole stirred. When the liquid has become clear, it is drawn off by a siphon, and a fresh quantity of sulphate of potash solution added. After some time the saline residue is transferred to a weighed filter, the funnel covered with a glass plate, and when the liquid has ceased to drop through, the filter with its contents is weighed, dried at 212° F., and again weighed. The difference between the two weighings gives the quantity of water evaporated from the sulphate of potash solution retained by the filter, and as the concentration of that solution is known, the quantity of sulphate of potash which it held in solution may be ascertained; the weight of this is deducted from the weight of the saline mass dried at 212° F. When the potash thus treated is free from soda, the remainder should be equal to the original weight of sulphate. But when soda is present in the potash, it is dissolved as sulphate, and the weight of salt after treatment with sulphate of potash solution is less than the original weight. The loss of weight in the latter case furnishes a means of calculating the per-centage of soda in the raw material. This loss of weight y bears the same proportion to the per-centage of soda x that the equivalent of sulphate of soda bears to the equivalent of carbonate of soda:

$$y : x = NaO,SO_3 : NaO,CO_2 = 71 : 53.$$

It must, however, be remembered that the soda used for adulterating potash would probably contain 20 per cent. of sulphate of soda. A correction must therefore be made when the amount of soda is large.

In making this experiment it is always desirable before proceeding to the weighings, to take the specific gravity of the sulphate of potash solution drawn off from the salt; if it is the same as before, there is no soda in the potash, but if sulphate of soda has been dissolved, the specific gravity is increased.

The marked difference in the reduction of temperature attending the solution of the alkaline chlorides in water, has been applied to the estimation of soda in potash.

a. When 50 grms. of chloride of potassium dissolve in 200 grms. of water, the temperature is reduced 11°·4 C. = 20°·52 F.

b. When 50 grms. chloride of sodium dissolve in 200 grms. of water, the reduction of temperature is only 1°·9 C. = 3°·42 F.

The reduction of temperature attending the solution of a mixture of both salts is dependent upon their relative proportion, and the per-centage of chloride of potassium x in the mixture may therefore be ascertained from the observed reduction of temperature y in any particular instance by means of the formula

$$x = \frac{100 \times y - 342}{17{\cdot}1},$$

in which y represents the difference between the temperature before and that after the solution of the saline mixture, x the per-centage of chloride of potassium, and z the amount of chloride of sodium.

About 60 grms. of the potash in which soda is to be estimated is neutralized with hydrochloric acid, the sulphuric acid separated by means of a normal solution of chloride of barium (p. 78), so as not to introduce any excess of baryta. The sulphate of baryta is collected upon a filter, and the clear filtrate containing only chloride of potassium and sodium, evaporated to dryness.

The experiment is made in a beaker-glass, weighing about 185 grms., and kept expressly for this purpose, so that the influence exercised by the cooling of the glass may be the same in all instances. 200 grms. of water are introduced into the beaker, the temperature observed, and 50 grms. of the dry residue thrown in. The whole is then stirred with the thermometer so that the solution of the salt may take place rapidly, and the lowest degree to which the temperature falls observed.

If the temperature of the water before adding the salt was 60°·8 F., and the lowest temperature reached during the solution 44°·6 F., then $y = 16{\cdot}2$, and

$$x = \left(\frac{100 \times 16{\cdot}2) - 342}{17{\cdot}1} = \frac{1278}{171} = 74{\cdot}7 \text{ per cent.}$$

The presence of chloride of calcium or magnesium in-

fluences the result so much as to render the method inapplicable in such cases.

Another method is based upon the solubility of the acetates of potash and soda in alcohol (0·83 sp. gr.), and the insolubility of perchlorate of potash in the same liquid; but it refers only to the soda present as carbonate.

The potash to be examined is first dissolved in water in such a proportion that 100 cubic centimeters represent the test quantity of material, and the usual alkalimetric experiment is then made with the normal acid; 10 cubic centimeters of the solution are then neutralized with acetic acid, the solution evaporated to dryness, and the residue treated with alcohol. Chlorides, sulphates, silicate, &c., remain undissolved; the solution, which will contain only acetates of potash and soda, is filtered into a beaker glass, and the quantity of soda estimated by means of a normal solution of perchlorate of soda in alcohol.

This normal solution is made by dissolving perchlorate of soda (NaO, ClO_7) in alcohol (0·83), in such proportion that 100 cubic centimeters contain 1·226 grm. of the salt. This quantity is just sufficient to precipitate as perchlorate 0·472 grm. KO (= 0·692 KO, CO_2); consequently if 10 cubic centimeters of potash solution—representing 0·472 grm. of the potash under examination—required for perfect precipitation 50 cubic centimeters of perchlorate solution, the potash would contain 50 per cent. of KO, and if the previous volumetric experiment with normal acid indicated a higher per-centage of alkali, the per-centage of soda in the state of carbonate may be calculated from the difference between the two results.

If, for instance, the quantity of normal acid required was 65 cubic centimeters, and the substance contains only 50 per cent. of potash, then 15 cubic centimeters were neutralized by the carbonate of soda, and as each cubic centimeter of normal acid is equivalent to 0·053 grm. NaO, CO_2, the carbonate of soda in the test quantity of potash will amount to

$$0{\cdot}53 \times 15 = 0{\cdot}795 \text{ grm. } NaO, CO_2,$$

and its per-centage x in the substance examined would be 16·8, for

$$16{\cdot}8 : 100 = 0{\cdot}795 : 4{\cdot}72.$$

Gravimetrical valuation of alkalies.

In the valuation of alkalies according to this method, the immediate experimental result is the quantity of carbonic

acid disengaged from a known quantity of the alkali by an excess of sulphuric acid. In order to ensure a sufficient degree of accuracy the quantity should not be less than 1 grm., nor for convenience sake more than 2 grms., and from it the quantity of alkaline carbonate x is ascertained by the proportion

$$x : c = \left\{\begin{matrix}\mathrm{KO,CO_2\ or}\\ \mathrm{NaO,CO_2}\end{matrix}\right\} : \mathrm{CO_2\ or}\ x = \frac{\left\{\begin{matrix}\mathrm{KO,CO_2\ or}\\ \mathrm{NaO,CO_2}\end{matrix}\right\}}{\mathrm{CO_2}} \times c.$$

If it is desired, the quantity of material experimented upon may likewise be regulated so that the quantity c expressed in centigrammes may give directly the per-centage of alkaline carbonate. Thus 6·92 grms. of carbonate of potash give 2·20 grms. of carbonic acid, and

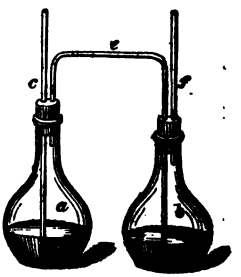
$$\begin{matrix}\text{centigrms.} & & \text{grms.}\\ 2{\cdot}20 : 6{\cdot}92 = 100 : 3{\cdot}14,\end{matrix}$$

5·30 grms. of carbonate of soda give 2·20 grms. of carbonic acid, and

$$\begin{matrix}\text{centigrms.} & & \text{grms.}\\ 2{\cdot}20 : 5{\cdot}30 = 100 : 2{\cdot}42;\end{matrix}$$

so that to obtain direct per-centage indications the quantity to be taken is, for carbonate of potash 3·14 grms., and for carbonate of soda 2·42 grms. When the per-centage of alkali is known to be small, or when the balance used is not sufficiently delicate to indicate a centigramme with certainty, it is advisable to take some multiple of these quantities, and to make a corresponding division of the result.

Fig. 33.



The apparatus used for this experiment (fig. 33) consists of two very light glass flasks, each fitted with tight corks, through which are inserted three tubes,—c extending to the bottom of one flask, f extending a little below the cork of the other flask, and a connecting tube e bent twice at right angles, and with one limb much longer than the other, as shown in the figure.

The experiment is made as follows. The flask b is one-third filled with concentrated sulphuric acid, and the weighed

quantity of alkali introduced into *a* together with enough water to dissolve it. The corks are then tightly fitted in, the upper part of the tube *c* closed with a plug of wax, and the whole apparatus weighed in this state. A piece of clean caoutchouc tube is then fitted upon *f*, and air sucked out by the mouth, until a few bubbles have passed through the sulphuric acid from the flask *a*, which is rendered air-tight by the wax plug; air is then allowed to enter the flask *b* through the caoutchouc tube, and a portion of sulphuric acid is thus forced over into the flask *a*, where it disengages carbonic acid from the alkali. This carbonic acid escapes by the tube *e*, and is dried in passing through the sulphuric acid in *b*. When the effervescence has ceased, more sulphuric acid is transferred to the flask *a* by sucking out air, and this repeated until a fresh addition of sulphuric acid no longer causes effervescence.

The heat generated during the action of the sulphuric acid is quite sufficient to expel all the carbonic acid from the liquid. The wax plug is loosened when the effervescence has ceased, air drawn through the flasks to displace the remaining carbonic acid, and the apparatus again weighed as soon as the contents of flask *a* have cooled. The difference between the two weighings gives the amount of carbonic acid, and, if the weights above indicated have been taken, the per-centage of carbonate in the substance examined.

Fig. 34.

A very much better form of apparatus for experiments of this kind (fig. 34) consists of a light vessel of blown glass with two bulbs *a* and *b*, connected at *d* by a short neck with a stop-cock, and by a tube *ccc* extending to the bottom of *b*. The bulbs are likewise furnished with necks at *i* and *h*, and below the stop-cock a piece of tube *e* extends to the bottom of the bulb *a*.

The substance to be examined is introduced into *a*, and the end of the tube *f* closed with wax. The bulb *b* is half

filled with sulphuric acid, a tube fitted by means of a cork into the neck *i*, and the whole weighed. Then, by opening the stop-cock *d*, acid flows into the bulb *a*, and as the tube *e* dips below the liquid, the gas generated can only escape through the tube *eee*, by which it is led into the sulphuric acid, and after traversing that, escapes dry through the tube *g*.

When this method of valuation is adopted, preliminary tests must be applied, in order to ascertain whether any substances are present which would influence the result. These are the following:—

Earthy carbonates, which may be removed by filtering the solution of the alkali in water.

Alkaline sulphurets, sulphites, and *hyposulphites*. The former disengages sulphuretted hydrogen, and the two latter sulphurous acid, which, if calculated as carbonic acid, would falsely augment the value of the alkali. This error may be prevented by mixing with alkalies which contain these substances a small quantity of yellow chromate of potash before making the experiment. By this means these substances are converted into sulphates.

Caustic alkali. When alkalies containing caustic alkali are valued by this method, the result obtained is too low, because it affords no indication of the caustic alkali present. The means of detecting the presence of caustic alkali is given above (p. 47).

To ascertain correctly by this method the value of a substance containing caustic alkali, this must first be converted into carbonate. The quantity weighed for an experiment is introduced into a porcelain capsule, mixed with 3 or 4 times as much clean washed quartz sand, and about $\frac{1}{2}$ or $\frac{1}{3}$ its weight of carbonate of ammonia, the whole moistened with water, dried gently, ignited to drive off the excess of carbonate of ammonia, and then transferred to the flask *a* in the usual manner.

When it is requisite to estimate the amount of caustic alkali as well as the carbonate, two experiments must be made; the first in the usual manner, and the second after converting the caustic alkali into carbonate as just described. The per-centage of caustic alkali x' may then be calculated from the difference. In the case of potash the proportion

would be as follows, supposing the difference between the quantities of carbonic acid found in the two experiments to be 17 :—

$$x : 17 = KO : CO_2; \text{ or } x = \frac{49{\cdot}2 \times 0{\cdot}17}{22} = 0{\cdot}381.$$

This 0·381 grm. KO would be contained in 6·29 grms. of the substance experimented upon, and the per-centage x' will be found by the proportion :—

$$x' : 100 = 0{\cdot}381 : 6{\cdot}29; \text{ or } x' = \frac{38{\cdot}100 \times 100}{6{\cdot}92} = 6{\cdot}05.$$

If the 17 centigrammes referred to soda, the first proportion would be,—

$$x : 17 = 31{\cdot}4 : 22; \text{ or } x = \frac{0{\cdot}17 \times 312}{22} = 0{\cdot}239;$$

and if 4·84 grms. of substance were taken, that for the per-centage x' would be,—

$$x' : 100 = 0{\cdot}239 : 4{\cdot}84; \text{ or } x' = \frac{239 \times 100}{4{\cdot}84} = 4{\cdot}73.$$

Acidimetry.

The per-centage value of acids may frequently be ascertained with tolerable accuracy by their physical characters, of density or boiling-point. The instruments used for this purpose, and the methods of operating, are described in the chapter on Hydrometry.

The chemical methods of valuation are precisely reverse applications of the principles upon which the value of alkalies is determined.

Volumetrical method of valuation for acids.

The reagent used may be either carbonate of soda, caustic soda, or caustic ammonia.

Of the former reagent 5·3 grms. neutralize quantities of the several acids indicated by the numbers below :—

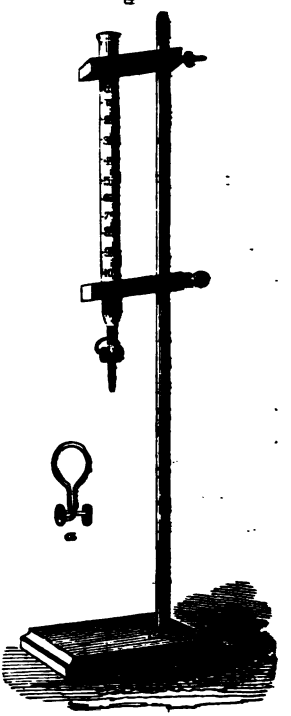
5·3 grms. NaO,CO_2 equivalent to	SO_3HO	4·9..	SO_3	4·0,
	NO_5HO	6·3..	NO_5	5·4,
	$\overline{A},HO$	6·0..	$\overline{A}$	5·1,
	ClH	3·65	Cl	3·55,
	PO_5HO	8·05	PO_5	7·13,
	$(C_8H_4O_{10}+2HO)$	15·0..	$(C_8H_4O_{10})$	13·2,
	$(C_{12}H_5O_{11}+4HO)$	20·1..	$(C_{12}H_5O_{11})$	16·5.

The normal solution is therefore made by dissolving 53 grms. of pure dry carbonate of soda in water to the volume of one litre; 100 cubic centimeters of this solution will neutralize the quantities of acid given in the table, and each cubic centimeter will contain $\frac{1}{1000}$ part of 53 grms. or 0·053 grm. NaO, CO_2.

The amount x of any acid represented by a certain volume of normal solution is found exactly in the same manner as in alkalimetry, by multiplying the number of cubic centimeters C_n by the quantity a of the particular acid to which a cubic centimeter of the normal solution is equivalent, the general formula being—

$$x = a \times C_n.$$

Fig. 35.



When the per-centage x' of acid is sought, it may be calculated from the quantity q operated upon, and the amount of acid x found, by the proportion,—

$$x' : 100 = x : q,$$

or it may be ascertained at once from the number of cubic centimeters of normal solution required for neutralization when q corresponds with the equivalent of the acid sought.

The quantity of material $= q$ to be taken in order to obtain direct per-centage results, is indicated for the several acids by the numbers in the table above; those in the first column referring to the per-centage of hydrated acid; those in the second column referring to the per-centage of anhydrous acid.

The experiment is made with the burette (fig. 35) in precisely the same manner as in alkalimetry, and resembles it, with the exception that the blue coloration of the litmus shows the end of the reaction. The evolution of carbonic acid is some

hindrance to the decisive indication of this point, inasmuch as it communicates a claret colour to the litmus, and owing to this circumstance, the addition of normal solution may be carried too far and the experiment spoiled. This inconvenience may be obviated in the case of acids that are not volatile by boiling the liquid after each addition of normal solution, and so expelling the carbonic acid.

When the acid to be examined is concentrated, it should be diluted with 3 or 4 times its volume of water. In the case of concentrated sulphuric or nitric acids, half the above quantities may be taken, and of dilute acids, appropriate multiple quantities, and a corresponding reduction of the results made.

The estimation of the acidity of wines is an exceptional case. For this purpose the normal solution must be diluted to 10 times its volume, and the quantity of wine cannot be weighed according to the equivalent of the acid sought.

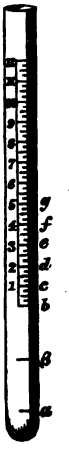
If in any experiment 100 cubic centimeters of wine require for neutralization 30 cubic centimeters of this decimal solution, this will correspond to 3 cubic centimeters of normal solution. This quantity will have been consumed in neutralizing two acids,—tartaric and acetic; the small quantity of citric acid which is usually present in wine may be disregarded. In order to ascertain the amount of each of these acids separately, another 100 cubic centimeters of the wine is evaporated to one-fourth on a water bath, by which means the acetic acid is expelled. The residue is then diluted somewhat and a second volumetrical experiment made, in which perhaps only 20 cubic centimeters of decimal solution are required for neutralization. This quantity corresponds to 2 cubic centimeters of normal solution, and represents the amount of tartaric acid present. The difference between the two results gives the quantity of acetic acid.

These quantities are calculated from the volume of normal solution requisite for neutralization. A cubic centimeter of normal solution (containing 0·053 grm. NaO,CO_2) is equivalent to 0·06 grm. of hydrated acetic acid ($\bar{A},HO$) and 0·15 grm. of crystallized tartaric acid. Consequently the 100 cubic centimeters of wine would contain—

Acetic acid..... 0·06 × 1 = 0·06 grm.
Tartaric acid ..... 0·15 × 2 = 0·30 „

In the valuation of vinegar likewise a decimal solution should be used, or 4 or 5 times the equivalent quantity for acetic acid taken, and the number of cubic centimeters of normal solution required for neutralization correspondingly divided, in order to obtain the per-centage value.

Fig. 36.



The valuation of vinegar may be effected with a normal solution of ammonia, containing 1·369 per cent. of the dry gas as the reagent. The apparatus required (fig. 36) is a tube closed at the bottom, about 12 inches long and 0·5 inch wide. The space below the mark *a* holds 1 grm. of water at 61·16° F. The space between *a* and *b* holds exactly 10 grms. of water at the same temperature. From *b* upwards the tube is graduated for degrees of 2·08 grms. of water each, or 2·07 grms. of ammonia solution of 1·369 per cent. Each of the 12 degrees is divided into four parts.

In making an experiment the tube is filled to *a* with litmus tincture, to *b* with the vinegar to be examined, and then the normal solution of ammonia added until the litmus becomes blue. The volume of normal solution used gives the per-centage of acetic acid ($\overline{A}$, HO). After each addition of acetic acid, which must be made in very small quantities, the tube is closed by the thumb, the contents shaken, and the thumb scraped along the edge so as not to remove any of the liquid.

When the vinegar is very strong, the tube is only filled to β, the remaining space between that point and *b* being made up with water, and the result doubled in order to obtain the per-centage value of the vinegar in acetic acid.

The accuracy of the results depends entirely upon the care with which the normal solution is prepared. The following table indicates the proportions of water and ammonia solution of different strengths to be used in making it.

Solution of ammonia, containing in 100 parts		has a density of	For 1000 parts of normal solution, containing 1·369 per cent. NH_3, there are required Solution of ammonia.	Water.
12,000	NH_3	0·9517	114·08	886·02
11,875		0·9521	115·3	884·7
11,750		0·9526	116·5	883·5
11,625		0·9531	117·8	882·2
11,500		0·9536	119·0	881·0
11,375		0·9540	120·0	880·0
11,250		0·9545	121·7	878·3
11,125		0·9550	123·0	877·0
11,000		0·9555	124·5	875·5
10,954		0·9556	125·0	875·0
10,875		0·9559	126·0	874·0
10,750		0·9564	127·3	872·7
10,625		0·9569	129·0	871·0
10,500		0·9574	130·4	869·6
10,375		0·9578	132·0	868·0
10,250		0·9583	133·5	866·5
10,125		0·9588	135·0	865·0
10,000		0·9593	137·0	863·0
9,875		0·9597	138·0	861·4
9,750		0·9602	140·4	859·6
9,625		0·9607	142·2	857·8
9,500		0·9612	144·0	856·0
9,375		0·9616	146·0	854·0
9,250		0·9621	148·0	852·0
9,125		0·9626	150·0	850·0
9,000		0·9631	152·0	848·0
8,875		0·9636	154·0	846·0
8,750		0·9641	156·4	843·6
8,625		0·9645	158·7	841·4
8,500		0·9650	161·0	839·0
8,375		0·9654	163·5	836·5
8,250		0·9659	166·0	834·0
8,125		0·9664	168·5	831·5
8,000		0·9669	171·0	829·0
7,875		0·9673	173·8	826·2
7,750		0·9678	176·6	823·4
7,625		0·9683	179·5	820·5
7,500		0·9688	182·5	817·5
7,375		0·9692	185·6	814·4
7,250		0·9697	188·8	811·2
7,125		0·9702	192·0	801·0
7,000		0·9707	195·6	804·4
6,875		0·9711	199·0	801·0
6,750		0·9716	202·8	797·2
6,625		0·9721	206·6	793·4
6,500		0·9726	210·6	789·4

Solution of ammonia, containing in 100 parts	has a density of	For 1000 parts of normal solution, containing 1·369 per cent. NH_3, there are required Solution of ammonia.	Water.
6,375 NH_3	0·9739	214·7	785·3
6,250	0·9735	219·0	781·0
6,125	0·9749	223·5	776·6
6,000	0·9745	228·0	772·0
5,875	0·9749	233·0	767·0
5,750	0·9754	238·0	762·0
5,625	0·9759	243·4	756·5
5,500	0·9764	249·0	751·0
5,375	0·9768	254·7	745·3
5,250	0·9773	260·8	739·2
5,125	0·9778	267·0	733·0
5,000	0·9783	273·8	726·2

An example will render the use of this table obvious. If, for instance, normal solution is to be prepared from ammonia solution of unknown strength, the density must be carefully estimated at a temperature of 61°·16 F. Supposing it to be 0·971, on referring to the table it will be found that this density corresponds to 6·875 per cent. of NH_3, and that 199 parts must be diluted with 801 parts of water in order to obtain normal solution containing 1·369 per cent. NH_3.

There is, however, a peculiar source of error in the estimation of acetic acid by neutralization, resulting from the retarding influence which alkaline acetates exercise upon the blue coloration of litmus. The difficulty may be obviated by the use of a solution of saccharate of lime as the reagent. This is prepared by igniting pure marble until the carbonic acid is expelled, mixing the slacked lime into a paste with water, and then adding pure sugar and water until the whole is dissolved. The liquid then contains from 50 to 56 per cent. of lime; it is diluted in such proportion that 100 cubic centimeters are exactly equivalent to 4·9 grms. of sulphuric acid (SO_3HO), or 6·0 grms. of acetic acid ($\bar{A}$, HO). It is well likewise to have a decimal solution, and both must be kept in well-stoppered vessels on account of the tendency to change by absorption of carbonic acid. In other respects the experiment is conducted the same as any other volumetrical estimation of acid.

The normal solution of carbonate of soda is perhaps the best in a practical point of view for acidimetric experiments that have a commercial or technical bearing, because it is

most easily prepared, and with some practice on the part of the operator very good results may be obtained with it.

But for estimations in which a higher degree of accuracy is desirable, the influence of the carbonic acid disengaged becomes a serious inconvenience. One of the various means of obviating this is to use instead of litmus a solution of pergallate of iron, which gives scarcely any tinge to the acid, but becomes intense violet colour with the minutest excess of alkali. This solution is prepared by adding to boiling dilute hydrochloric acid, an excess of the bluish black precipitate, formed by mixing gallic acid with a neutral solution of perchloride of iron, and separating the undissolved portion by filtration.

The plan, however, which most fully ensures a high degree of accuracy in the results is the use of a normal solution of caustic alkali, which, to prevent the absorption of carbonic acid, is kept in a glass vessel, closed by a tight cork, through which passes a bulbed tube, containing a mixture of sulphate of soda and caustic lime. A free communication is thus maintained between the air within and without the bottle, while the access of carbonic acid is perfectly prevented.

Gravimetrical method of valuation for acids.

This method is based upon the fact that all the stronger acids readily and completely decompose an equivalent weight of carbonate of soda, so that when any acid is added to an excess of carbonate of soda it is easy to ascertain the amount of real acid x in the substance from the quantity of carbonic acid c disengaged, by the proportion

$$x : c = \text{acid} : CO_2;$$

and when the quantity q operated upon, whether weight or volume, is known, the per-centage x' will be

$$x' : 100 = x : q.$$

It is necessary to use bicarbonate of soda for experiments by this method, because when an acid is added to an excess (which there must necessarily be) of neutral carbonate, the full equivalent of carbonic acid is not disengaged, a portion being retained by the excess of carbonate in the form of bicarbonate, and the consequence of this would be that the results obtained would be always too low.

The trouble of calculation may be avoided by taking for the experiments such quantities of the acids, that the quantity of disengaged carbonic acid expressed in centigrammes will give at once the per-centage of real acid in the substance examined.

Here, as in alkalimetric experiments by this method, the quantity of carbonic acid disengaged should not be less than 1 grm., nor more than 2 grms. 44 parts of carbonic acid are disengaged from bicarbonate of soda by 40 parts of SO_3, 49 parts SO_3HO, or an equivalent quantity of any other acid, and the quantities x of any acid which are equivalent to 100 centigrammes of CO_2 are shown in the following table:—

grms.		grms.			centigrms.			grms.
44 CO_2	:	40	(SO_3)	=	100	:	x	(= 9·1 SO_3).
„	:	54	(NO_5)	=	„	:	„	(= 12·3 NO_5).
„	:	36·5	(HCl)	=	„	:	„	(= 8·3 HCl).
„	:	51	($\bar{A}$)	=	„	:	„	(= 11·6 $\bar{A}$).
„	:	132	($\bar{T}$)	=	„	:	„	(= 30·0 $\bar{T}$).
„	:	165	($\bar{C}$)	=	„	:	„	(= 37·5 $\bar{C}$).

The numbers in the latter columns indicate the quantities of substance to be taken in order to ascertain directly the per-centage of anhydrous acid.

If 123 centigrammes of a sample of nitric acid is found to disengage 50 centigrammes of carbonic acid, it will contain 50 per cent. of NO_5.

As most of the acids met with in commerce are more or less dilute, it will sometimes be necessary to take multiples of the above quantities, and to divide the result obtained.

Thus, if in the examination of nitric acid 2·46 grms. give 40 centigrammes CO_2, then the per-centage of NO_5 would be 20.

If 69·6 grms. (60×116) of vinegar gives in experiment 180 centigrammes CO_2; then as $\frac{180}{60} = 3$, it would contain 3 per cent. of anhydrous acetic acid.

In the valuation of acids by this method, as well as by the volumetrical method, preliminary experiments must always be made for the purpose of ascertaining whether they are free from any substances which would act in the same manner as the acid sought to be estimated, and so lead to a

false result. Thus, when nitric acid contains hydrochloric or sulphuric acid, the per-centage result obtained for NO_5 would not only be too high, but, inasmuch as the equivalents of these acids are about two-thirds that of NO_5, 2 per cent. of these impurities will be taken as 3 per cent. of NO_5. In such cases the valuation experiment becomes more complicated. The result will therefore require correction, which may be made by estimating the quantity of sulphuric hydrochloric acid or other foreign acid $= z$, in the quantity experimented upon, either by means of suitable normal solutions, or by weighing the precipitates, and then calculating the quantity of $CO_2 = c'$ that would be disengaged by it from $NaO, 2CO_2$, by the proportion,—

$$z : c' = SO_3 : 2CO_2 \text{ or } z : c' = HCl : 2CO_2.$$

The value thus obtained for c' is then to be deducted from the result obtained in the first experiment; the remainder gives the true per-centage of NO_5 in the acid examined.

Fig. 37.

The apparatus used is the same as that for the valuation of alkalies, but it is necessary to have a small glass tube that will pass through the neck of the flask for containing the carbonate of soda. This tube is furnished with a small piece of very thin platinum wire (fig. 37).

The experiment is also made much in the same manner. The acid to be examined is introduced into the flask *a*, if necessary diluted, so as to make up about 30 or 40 grms., and the small tube, filled with dry bicarbonate of soda, is suspended in the flask by means of the wire which is clamped between the cork and the neck of the flask, so that the acid may be brought in contact with its contents by a slight inclination of the apparatus. In this state the apparatus is weighed, and the acid then brought in contact with the bicarbonate. The carbonic acid disengaged is dried by passing through the sulphuric acid in *b*, and when the reaction is completed, the flask *a* is placed in water of about 160° F. to expel carbonic acid from the liquid, and the residual atmosphere of the gas in the

flasks removed by loosening the wax plug in *c* and drawing air through them. The apparatus is then dried, allowed to cool, and again weighed. The difference between the two weighings gives the amount of carbonic acid disengaged, and when the quantities of substance taken for the experiment are those given in the table, this difference, expressed in centigrammes, gives the per-centage of acid sought.

Estimation of acids in combination with bases, and valuation of the saline compounds of the alkalies. (*Halimetry.*)

The valuation of saline compounds of the alkalies is generally effected by estimating the amount of the acid they contain by means of some reaction that admits of being adapted to the volumetrical method. In some instances the valuation must be effected by means of some physical peculiarity of the substance sought.

Estimation of sulphuric acid.

The precipitation of sulphuric acid, as sulphate of baryta (BaO, SO_3), by soluble baryta salts may be applied to the estimation of this acid in soluble compounds. According to the equation—

$$BaO, SO_3,$$

4 parts of sulphuric acid (SO_3) are equivalent to 7·65 baryta (BaO), and the normal solution may be made by dissolving 122 grms. of chloride of barium in water to the volume of one litre; each cubic centimeter will then be equivalent to 0·04 grms. of sulphuric acid (SO_3).

The amount of sulphuric acid may then be found by multiplying the number of cubic centimeters required for perfect precipitation by 0·04.

The very tardy separation of the sulphate of baryta precipitate is, however, a serious obstacle to the application of this process. In some instances it may be the only one applicable, and then it would be better to collect and estimate the sulphate of baryta by weighing; the amount of sulphuric acid, or of the sulphate sought being calculated in accordance with the above formula and that of the salt. In the absence of a balance or other conveniences for the purpose, recourse must be had, in the volumetrical experiment,

to the application of heat to the liquid in which the sulphate of baryta is suspended, or filtration.

A much better mode of estimation consists in precipitating the acid, as sulphate of lead (PbO, SO_3), by nitrate of lead: and the reaction between iodide of lead and alkaline sulphates,

$$KO, SO_3 + PbI = KI + PbO, SO_3,$$

furnishes a very marked indication of perfect precipitation; so that when the liquid in which sulphuric acid is to be estimated is mixed with a few drops of iodide of potassium solution, there is, on the subsequent addition of the normal solution of nitrate of lead, no *permanent* formation of iodide of lead until the whole of the sulphuric acid has been removed from solution, and then the yellow colour of the precipitate does not disappear on shaking the mixture.

The normal solution is made by dissolving 41·33 grms. of nitrate of lead in water to the volume of one litre, and the results are calculated as in the case of the baryta solution, each cubic centimeter being equivalent to 0·010 grm. SO_3.

The absence of any substances which might be precipitated, like sulphuric acid by the normal solution, must always be ascertained by appropriate tests.

Estimation of nitric acid.

The conversion of protochloride of iron (Fe Cl) into perchloride ($Fe_2 Cl_3$) by nitric acid, together with the estimation of iron by permanganate of potash, has been applied to this purpose. The reaction is—

$$6Fe\,Cl + NO_5 + 3HCl = 3Fe_2 Cl_3 + NO_2 + 3HO.$$

The amount of nitric acid x sought is then calculated from the quantity of iron F converted into perchloride by the proportion—

$$x : F = NO_5 : Fe_6.$$

Valuation of nitre.

For the manufacture of gunpowder it is of great importance to be able to estimate the amount of impurities in the commercial nitrate of potash by some brief and easy operation.

The method followed in France depends upon the fact that a solution of nitrate of potash, saturated for a certain temperature, is incapable of dissolving any more of the salt, although it will dissolve the other salts with which it is usually contaminated.

For testing a sample of nitrate, 400 grms. of the powdered salt are mixed with 500 cubic centimeters of a saturated solution of pure nitrate in a bottle and well shaken for 10 or 15 minutes, the liquid poured off into a filter, the remaining salt agitated with 250 cubic centimeters more of the pure nitrate solution, and the whole thrown upon the filter. When the solution has passed through, the salt is freed as much as possible from adherent liquid by pressure against bibulous paper, completely removed from the filter, dried, and weighed. The loss of weight which it is found to have suffered in this operation represents the quantity of impurities present in the nitrate.

It has, however, been ascertained that a serious source of error attaches to this method of valuation. It consists in the circumstance that a saturated solution of nitrate becomes capable of dissolving an additional quantity on the addition of chloride of sodium, and that the quantity so dissolved and the consequent error is greater in proportion to the amount of chloride of sodium present. The presence of chloride of potassium likewise involves an error in the opposite direction, and this method of valuation should not be adopted without very great precaution.

Another method has been adopted in Austria. It depends upon the decrease of the solubility of nitrate of potash in water consequent upon reduction of temperature, or, in other words, the more or less speedy separation of solid nitrate from a hot solution containing a known quantity of crude nitrate when allowed to cool. The temperature at which the first signs of crystallization appear is taken as an indication of the per-centage of pure nitrate. The disadvantage attending this method is, that the point of saturation for a given temperature varies according to the amount of chloride of sodium present. There are likewise other sources of error attached to this method, and the possible variation in the results it furnishes are too great to admit of its being considered satisfactory.

In Sweden the appearance of the fracture of the salt after melting is taken as an indication of its value. The fracture of the pure nitrate has a lustrous, radiated, crystalline appearance; but when chloride of sodium is present, to the amount of 2·5 per cent., the fracture is dull, more granular, and com-

pact. The intermediate gradations, however, are so little characteristic, that it is hardly possible by their aid to form any trustworthy opinion as to the per-centage of chloride in particular specimens.

The chemical methods of valuation are far more accurate. One is based upon the convertibility of nitrate of potash into carbonate by ignition with carbon, and the fact that chloride of sodium or potassium are not affected under the same conditions. By estimating the quantity of carbonate by a given weight of crude nitre, it is therefore possible to calculate its per-centage of pure nitrate.

The ignition must be made with some precaution, on account of the violent action that takes place, to moderate which it is advisable to add 3 or 4 times as much chloride of sodium, and to project the mixture in small portions into a red-hot iron crucible. When the reaction is at an end the crucible is allowed to cool, the contents dissolved in water, and the volume of the filtered solution made up to 200 cubic centimeters. The estimation of the carbonate of potash is made as directed at p. 56. Unfortunately nitrate of soda is converted into carbonate by the same treatment, and when alkaline sulphates are present, they are converted into sulphurets, which likewise affects the result, so that this method is inapplicable to the valuation of nitre containing these salts, unless at the same time other experiments are made to ascertain their amount.

The process already described for the estimation of nitric acid (p. 79) may in most instances be applied to the valuation of nitre. The experiment is made in the manner there described, and the amount of nitrate of potash x sought, calculated from the quantity of iron F converted into perchloride by the proportion:—

$$x : F = KONO_5 : Fe_6.$$

Experiment. Two grammes of clean iron wire are dissolved by hydrochloric acid in a flask of 200 cubic centimeters' capacity, with a narrow neck, closed by a perforated cork, through which passes a glass tube, drawn out to a small bore for the escape of gas. To the solution of protochloride of iron, containing excess of acid, is added 1·2 grm. of the crude nitre, the flask closed, and the whole heated to boiling. The greater the per-centage of nitrate in the nitre, the greater

the quantity of perchloride of iron formed. Chloride of potassium or sodium and sulphates do not affect the result.

It remains then to ascertain how much iron F has been converted into perchloride. For this purpose the normal solution of permanganate of potash is used precisely in the same manner as for the estimation of iron (Chap. VII.). The solution is used of such value that 50 cubic centimeters are sufficient to convert 1 grm. of iron, in the state of protochloride, into perchloride, and it thus indicates the amount of iron F still remaining in the state of protochloride. 2 grms. of iron in the state of protochloride require for conversion into perchloride 1·216 grm. of pure nitrate of potash.

If in a particular experiment 10 cubic centimeters of normal permanganate solution suffice to complete the formation of perchloride of iron after the reaction of the protochloride and nitrate, the quantity of iron F converted into perchloride by the nitre is 2 grms. minus the quantity of iron F, equivalent to this volume or 1·8 grm., and the quantity of pure nitrate x requisite for this purpose is ascertained by the proportion:—

$$2{\cdot}00 : 1{\cdot}216 = 1{\cdot}80 : y \quad (y = 1{\cdot}0944).$$

Then, as 1·2 grm. of nitre was used, its per-centage of pure nitrate x' is found thus:—

$$1{\cdot}2 : 1{\cdot}0944 = 1{\cdot}00 : x \quad (x = 91{\cdot}2 \text{ per cent.}).$$

The per-centage of sulphates or chlorides may be estimated by means of normal solutions of baryta or silver salts, or by the methods described at p. 79 and Chap. VIII.

It has been found, by the examination of a great number of samples of crude nitre, that the proportion of the chlorides of potassium and sodium is one Na Cl to two KCl, and the per-centage of each chloride may be calculated from the quantity of chlorine found in accordance with this result.

In the case of East Indian nitre at least, it is more important to estimate the per-centage of alkaline earths rather than that of sulphates, which is very small. For this purpose lime and magnesia may be precipitated by carbonate of soda, and estimated as described in Chap. IX. In most instances the per-centage of magnesia is nearly as great as that of lime, and no great error is made by regarding the precipitate as half lime and half magnesia.

None of these methods give any indication of the per-

centage of nitrate of soda. Except as an adulteration it is rarely present in East Indian nitre, but some kinds of nitre contain a considerable amount, for ascertaining which there is no trustworthy means besides the direct estimation of the potash. The fact that nitrate of soda attracts moisture from the atmosphere, is had recourse to for an approximative estimation of this salt. Pure nitrate of potash exposed for 14 days under a bell-jar with a basin of water attracts no moisture, but when nitrate of soda is mixed with it an increase of weight is observed.

Per-centage of nitrate of soda. . . .	0·5, 1, 3, 5, 10.
Increase of weight in 14 days. . . .	2·5, 4, 10, 12, 19.

Analysis of gunpowder.

This operation is tedious and difficult when it is necessary to separate perfectly the several ingredients.

The first step is to estimate the *amount of moisture* by exposing 5 or 6 grms. of the powder under a bell-jar with a basin of sulphuric acid for some days, and weighing several times, until no further decrease of weight takes place.

The per-centage of nitrate of potash is ascertained by treating about 10 grms. of the powder with warm water. The insoluble residue, consisting of sulphur and carbon, is washed upon a weighed filter, dried and weighed. The difference in the weight gives the amount of nitrate of potash, which may be confirmed by evaporating the filtrate to dryness and weighing the saline residue.

The estimation of the sulphur is effected by melting 1 or 2 grms. of the powder with equal quantities of pure carbonate of soda and nitrate of potash, and four times as much chloride of sodium, until the whole becomes white. The carbon is thus converted into carbonic acid, the sulphur into sulphuric acid. The fused mass is dissolved in water, treated with an excess of hydrochloric acid, and the sulphuric acid estimated either by weighing as sulphate of baryta, or by means of a normal solution of chloride of barium, one cubic centimeter of which is equivalent to 0·004 grm. SO_3. If therefore the sulphuric acid y yielded by 1 grm. of gunpowder requires for precipitation, as BaO,SO_3, 62·5 cubic centimeters of normal solution, it will amount to $4 \times 62{\cdot}5 = {\cdot}250$ grm.,

and the quantity of sulphur x represented by it may be ascertained by the proportion:—

$$x : y = S : SO_3, \text{ or as } 16 : 40;$$

accordingly $x = 100$ grm., and the powder would contain 10 per cent. of sulphur.

The per-centage of carbon may be ascertained by deducting from the weight of powder operated upon, the sum of the quantities of moisture, nitrate of potash and sulphur, found, and calculating for 100 parts.

When, as is sometimes the case, it is desirable to ascertain what kind of charcoal has been used for the preparation of gunpowder, the separation of the sulphur must be effected in a different manner. A weighed quantity of the mixture of sulphur and carbon is digested for some time with a solution of sulphuret of carbon in absolute alcohol, by which means the sulphur is dissolved, and the carbon left unaltered.

It may likewise be necessary to estimate sand and similar admixtures, which is done by washing out the nitrate, burning off the sulphur and carbon, and weighing the residue. This residue will, however, contain the fixed substances originally contained in the sulphur and charcoal.

The compositions used for fireworks are analogous in composition to gunpowder. They may be classified as—

a. Mixtures containing nitre with charcoal, sulphur, organic substances (rosin, glue), and salts which colour the flame.

b. Mixtures containing chlorate of potash with sulphur, charcoal, and sometimes sulphuret of antimony, or other substances for colouring the flame.

The first group comprises the gray composition, and the firework charges prepared from it and from meal powder. The second group comprises the charges used for signal rockets and fusees.

Compositions containing nitre may be analysed in the same manner as gunpowder, but the operation is rendered somewhat more difficult by the presence of the organic substances, which it is difficult to estimate, and which even hinder the accurate estimation of the other ingredients.

Colophony is a frequent ingredient of these compositions; it is separated most conveniently by boiling a weighed

quantity of the substance with alcohol of 0·81 sp. gr., evaporating the filtered solution, and weighing the dry residue.

Glue or *gum* must be separated by water, which likewise takes up the soluble salts. When it is desired to estimate these substances, the solution is evaporated to dryness, weighed, ignited in a crucible treated with hydrochloric acid, the solution again evaporated to dryness, ignited, and the quantity of nitrate of potash calculated from the chloride of potassium found (100 parts of KCl represent 134 KO,NO_5). The difference between the calculated weight of nitrate and the total weight of the substances dissolved out by water, gives the amount of organic substance.

Sulphuret of antimony is easily detected by the glittering appearance of the portion insoluble in water, and by its density. It is estimated by boiling the residue, left after separating nitre, with strong hydrochloric acid, treating the filtered solution with sulphuret of hydrogen, collecting and weighing the precipitate of sulphuret of antimony.

The most important of the compositions containing chlorate of potash is that used for friction fusees. This consists of varying quantities of sulphuret of antimony with chlorate of potash, agglutinated by a little isinglass.

The chlorate of potash is estimated by treating the mass with water, gently warming, and after the addition of a few drops of infusion of galls, filtering. The filtrate contains, after the insoluble residue has been washed, all the chlorate of potash; it is evaporated to dryness, ignited, mixed with a little hydrochloric acid, again dried, ignited, and the quantity of chlorate calculated from the chloride of potassium obtained (100 KCl represent 164 KO,ClO_5). The difference between this calculated weight and that of the substance operated upon gives the quantity of sulphuret of antimony. The cementing substances never amount to more than 0·25 or 0·50 per cent.

The mixtures for coloured fires containing nitrate of baryta, sulphate of copper and ammonia, nitrate of strontia and sulphuret of antimony, likewise contain, besides these substances which produce the colour, sulphur, carbon, and chlorate of potash. Their analysis is conducted by separating in the first instance the soluble from the insoluble ingredients. Baryta is separated and weighed as sulphate (100 BaO,SO_3

represent 111·5 BaO, NO_5); the filtrate containing excess of sulphuric acid, leaves on evaporation sulphate of potash, from the quantity of which the quantity of chlorate of potash is calculated (100 KO, SO_3 represent 140 KO, ClO_5). Mixtures containing strontia are treated in exactly the same manner (100 SrO, SO_3 represent 115 SrO, NO_5). Copper is estimated in the solution from cupreous mixtures by treatment with sulphuretted hydrogen, collecting the precipitate, which is washed with solution of sulphuretted hydrogen, dried and ignited, first alone, and then with carbonate of ammonia until its weight remains constant. The quantity of sulphate of copper and ammonia is calculated from the weight of oxide of copper found (100 CuO represent 307 of the salt).

The insoluble portion of these mixtures is either charcoal, sulphur and charcoal, or these ingredients together with sulphuret of antimony; they are estimated in the manner described above.

It is sometimes necessary in the first instance to make a qualitative examination of such substances according to the system described in Chapter X.

Valuation of borax.

Borax has a slight alkaline reaction upon litmus, while free boracic acid colours it like carbonic acid, and the red colour is produced only when there is an excess of some stronger acid, as sulphuric acid. Consequently the normal solution used in alkalimetry may be applied to the valuation of borax, and in the same manner.

The quantity of the crystallized salt to be taken for an experiment is 19·08 grms., and from the volume of normal solution requisite to produce the red colour 0·5 cubic centimeter is to be deducted, because the presence of sulphate of potash retards the action of the acid.

CHAPTER VI.

THE ALKALINE AND TRUE EARTHS, AND THEIR COMPOUNDS; THE TESTS OF THEIR PURITY, AND METHODS OF VALUATION, ETC.

Barium compounds.

Caustic baryta, chloride of barium, nitrate of baryta, and *carbonate of baryta* are employed as chemical reagents for a variety of purposes. *Chlorate of baryta* is used in the preparation of fireworks, and *sulphuret of barium* for separating arsenic and nitrogen acids from sulphuric acid. The *native sulphate of baryta* is used as a pigment, either alone or mixed with others.

Sulphate of baryta is rarely free from oxide of iron, strontia, and lime, and as it is the source from which all baryta compounds are derived, these impurities may probably be present in them.

Iron is indicated by a blue precipitate with ferrocyanide of potassium.

Strontia is indicated by the carmine colour which its chloride communicates to the flame of alcohol. In testing the substances 1, 3, 4, 5, and 6, they are treated with hydrochloric acid, and evaporated to dryness; sulphate of baryta is boiled with a solution of carbonate of soda, the liquid filtered hot, neutralized with hydrochloric acid, and evaporated to dryness. The dry residue of chloride obtained in each case is digested for some time with strong alcohol, which is ignited in a basin after filtration.

Lime is indicated by the solubility of its sulphate and by a white precipitate with oxalate of ammonia in the neutral solution, from which baryta has been precipitated as sulphate.

Heavy metals are indicated in soluble baryta compounds by a precipitate with sulphuretted hydrogen, in the presence of excess of hydrochloric acid.

The above-mentioned barium compounds should be per-

fectly white, with the exception of the sulphuret, which has a yellow tinge; 1, 2, 3, and 5 should be perfectly soluble in water, 4 in hydrochloric, nitric, or acetic acids. The contamination of one baryta salt by another is indicated by the test for the corresponding acid.

The most distinctive character of soluble barium compounds is the formation of an insoluble compound when treated with sulphuric acid, or a soluble sulphate, and the perfect insolubility of this compound (BaO, SO_3) in water or acids.

Strontium compounds.

Chloride and **nitrate** of strontium are used in the preparation of coloured fires and fireworks. They much resemble the corresponding barium compounds.

The most probable impurities are,—

Barium or *calcium salts.* They are indicated by a precipitate on the addition of a few drops of ferrocyanide of potassium to a moderately strong solution of the salt. Calcium may be especially tested for in the same way as in barium compounds.

The distinctive character of soluble strontium compounds is, that when mixed with burning alcohol they communicate to the flame a carmine colour.

Calcium compounds.

Carbonate of lime, CaO, CO_2, and **caustic lime, CaO.**

Lime, as it is used for various purposes in the arts, is obtained by igniting, in a suitable furnace, carbonate of lime in the form either of limestone, chalk, or marble, and the process has been named after the product, calcination.

All these materials are more or less impure, and their value for the preparation of lime depends greatly upon the nature and amount of foreign substances.

Among the very numerous uses of lime in the arts, there are some for which it is necessary that the lime should be pure, while for others the presence of certain other substances is even advantageous. Thus in the preparation of caustic alkalies, and other chemical operations, as well as in soap-making, bleaching, dyeing, tanning, sugar-refining, purification of gas, and glass-making, the lime used should

be as pure as possible. For the preparation of mortar, plaster, and other building purposes, the purity of the lime is less essential, and for the preparation of cements, certain kinds of lime containing other substances are preferable.

Lime obtained by calcining a tolerably pure limestone should, when moistened with a little water readily crumble down with considerable evolution of heat, or as it is called, slake, and when mixed with more water form a smooth paste.

Lime containing a considerable proportion of foreign substances—clay, magnesia, peroxide of iron, &c.—slakes less readily, absorbs less water, and forms a granular paste.

The difficulty in slaking may, however, be owing to defective calcination as well as to the presence of foreign substances.

Different kinds of lime vary in other respects in their behaviour with water; some kinds of lime have the peculiar property of becoming perfectly compact and hard after a time under water. This is called hydraulic lime. The hardening is owing to the presence of a certain proportion of aluminous, siliceous, and alkaline substances.

Cements are variable mixtures of lime with siliceous or argillaceous materials, which communicate to it the property of hardening under water.

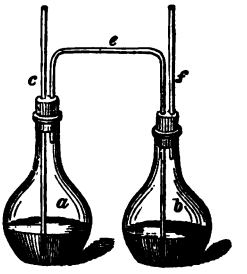
The value of lime, carbonate of lime, marl, &c. for various purposes is generally ascertained in the same manner. When it is necessary that the examination should be comprehensive, the method described in Chapter IX. must be adopted, but for most practical purposes the following experiment, referring more specially to lime, will afford sufficient indication of the essential characters of any of these materials.

1. *Water* is detected and estimated in lime, limestone, marl, &c., by igniting a weighed quantity in a porcelain crucible, and again weighing. When the two latter contain organic matter, the loss of weight will represent both it and water. For the estimation of water alone a temperature of 212° or 250° F. must be applied.

2. *Carbonic acid* may be present in a sample of lime either from imperfect calcination, or in consequence of absorption from the atmosphere when the lime has been long kept. It is at once indicated by effervescence on the addition of an acid. The carbonic acid in limestone or lime is estimated

by means of the apparatus (fig. 38), in the same manner as the value of an alkali.

Fig. 38.



3. Limestone frequently contains *organic substance*. It is generally evident from the dark colour of the mineral, and is estimated by strongly igniting a weighed quantity of the powdered limestone, previously dried at 212° F., then moistening it with a few drops of carbonate of ammonia solution, drying, gently igniting, and weighing the residue. The loss of weight gives the amount of organic substance with sufficient accuracy for all practical purposes.

4. *Silica, alumina, oxide of iron, magnesia,* and *alkalies.* The various kinds of native carbonate of lime, and consequently caustic lime, are seldom free from one or more of these substances in greater or less proportion, sometimes associated with very small quantities of phosphoric and sulphuric acids.

The partial insolubility of some limestone in dilute acids is chiefly owing to the presence of silica and alumina. These substances may, however, exist in limestone, &c. in two different states,—in the one case soluble in acids, in the other insoluble; but, generally speaking, the silica and alumina in limestone, &c. is, for the most part at least, insoluble in acids, while in the caustic lime they are soluble in acids, the calcination having converted them into the state of soluble compounds with lime.

The question whether a particular kind of limestone would be suitable, when calcined, for the preparation of mortar, was for a long time determined by the chemical characters, according to a simple method proposed by Vicat, a very high authority on the subject of hydraulic lime.

Limestone which contains argillaceous ingredients may be regarded as a mixture of carbonate of lime and silicate of alumina (clay), in which the latter exists in very variable proportion. As carbonate of lime (also carbonate of magnesia) is readily soluble in dilute hydrochloric acid, while the argillaceous portion is for the most part insoluble in the

same reagent, the hydraulic property of a sample of limestone was estimated according to the amount of insoluble residue left after treatment with dilute hydrochloric acid. This was collected on a filter, washed, dried, ignited, and weighed.

This method, which is still adopted, is, however, very imperfect, and altogether inadequate for the purpose to which it is applied. Vicat has himself pointed out that it is only capable of furnishing very rough approximative indications, and that it is by no means sufficient to estimate merely the amount of ingredients insoluble in hydrochloric acid, since their influence upon the value of limestone for mortar may be of a twofold nature. One portion of them is by the process of calcination rendered soluble by the aid of hydrochloric acid, as is shown by the separation of gelatinous silica on evaporating the hydrochloric solution of the lime, and by the formation of a hard mass insoluble in water when the lime is mixed with water. The other portion of the insoluble ingredients consists of mechanical admixtures, frequent coarse granules of quartz sand, and fragments of disintegrated siliceous minerals.

It is therefore far preferable to calcine the limestone before applying Vicat's test, because it is then at once seen how much of the ingredients, insoluble in hydrochloric acid, are converted into the soluble state by calcination. When the time and other requisites for a complete chemical analysis of the limestone are wanting, the following plan may be adopted as likely to furnish some useful indications :—

Several fragments of three or four inches diameter, and selected as representing the average character of the material, are broken into two pieces, one of each being introduced into a large earthen crucible with holes bored through the bottom, and exposed to a suitable temperature for calcination, either in a lime-kiln or in the laboratory furnace (fig. 17).

The other pieces are powdered and thoroughly mixed. A weighed quantity, 2 or 3 grms., is reduced to a very fine state of division in an agate mortar, dried at 212° F., and treated with moderately dilute hydrochloric acid at a gentle heat. The insoluble residue is collected on a filter, washed, dried, and weighed.

The calcined portion is treated in exactly the same manner, but to render the results comparable an allowance

must be made for the loss of weight by calcination. This is best done by weighing the limestone calcined both before and after calcination. If, for instance, the loss amounted to 35 per cent., then 65 parts of calcined lime would represent 100 parts of the limestone, and the residue, insoluble in hydrochloric acid, from that quantity would be equal to the mechanical ingredients of 100 parts of limestone.

The necessity for calculation may be avoided by taking for the experiment 3·00 grms. of the raw material, and of the calcined lime 3 times as many centigrammes as there are hundredths of lime yielded by the limestone when calcined. Thus, in the above instance, there would be taken as equivalent to 3·00 of raw material 3 × 65 centigrammes = 1·95 grm. The insoluble residue from that quantity would represent the portion not rendered soluble by calcination, and the mechanical admixtures of 3·00 grms. of raw material, and the percentage would be obtained by division of that quantity by 3.

In examinations of this kind a repetition of the experiment is always desirable, in consequence of the variation in the chemical character of limestone, and of the differences arising from dissimilar calcination.

Alkalies. It has been satisfactorily proved that the presence of alkalies in calcined argillaceous lime has a very great influence upon its hydraulic properties, and it is one of the chief defects of Vicat's method that it gives no indications with reference to this particular.

The alkalies may, as well as silica and alumina, exist partly in the soluble and partly in the insoluble state. The marls of looser texture are always mixed with disintegrated fragments of rock, which, although they resist the action of acids, even after calcination, still contain alkalies. Since, however, such ingredients contribute little, if at all, to the hydraulic character of lime, it would be exceeding the limits of practical inquiry to direct attention to them any further than to include them under the head of insoluble ingredients.

That portion of the alkalies, however, which is rendered soluble by calcination is of considerable importance. It may be estimated with sufficient accuracy by the method proposed by Fehling and Faisst:—40 or 50 grms. of the calcined lime are mixed with water in a capsule (lime which hardens with water should be mixed with solution of car-

bonic acid), the whole allowed to rest for awhile, the clear liquid poured off, and a stream of carbonic acid passed through it to separate the lime; the remaining liquid evaporated to dryness and dissolved in water, filtered, and the residue obtained by evaporating the clear filtrate, weighed as carbonate of potash and soda with traces of chloride of sodium.

Another chemical test for ascertaining the relative value of cements, which gives, however, no indication as to their composition, depends upon their power of precipitating lime from its aqueous solution. Weighed quantities of the cements are taken in fine powder and gradually mixed with equal quantities of lime-water, constantly stirred meanwhile, and the addition continued until the whole of the lime is removed from solution, which may be ascertained by testing with carbonate of soda. The remainder of each sample of cement is then weighed, and the value of the cement estimated as inversely proportional to the quantity required to precipitate a certain quantity of lime.

The quality of a sample of cement or hydraulic lime may also be estimated by trials as to the time taken in hardening, and the degree of hardness acquired. This test should never be omitted, even when a complete chemical analysis is made.

It may happen that a native or fabricated mixture of argillaceous and calcareous ingredients, which corresponds fully with all the requisites for the production of a good cement or hydraulic lime, will, after calcination, when exposed for a long time to atmospheric influences, become so altered as to be wholly valueless as a cement.

When such an alteration has merely amounted to the absorption of carbonic acid, repeated calcination will restore the value of the material; but the absorption of water, however, has a further prejudicial effect, which is not so easily remedied, for the change which renders the material useful as a cement takes place—the siliceous ingredients and the lime combine, forming a hydrated silicate; and even when this is only partially the case, the property of hardening when mixed with water is diminished in a corresponding degree.

The particulars to be observed in testing cement with a view to the property of hardening are described by Pasley and Schafhäutl, as follows:—

1. The cement in very fine powder is mixed with just enough water to make it into balls about an inch in diameter. During the absorption of water these balls should become warm, but not positively hot. When they have cooled, (which, with good cement, will be in about half an hour,) they are laid in a vessel of water, and when they continue to become harder, and after the lapse of a few days are perfectly hard throughout the entire mass, the cement may be considered good, and the contrary when the balls have not acquired a considerable degree of hardness.

2. When the balls of cement do not become hard under water, it may be desirable to ascertain whether it is owing to the natural inferior quality of the material, to deterioration by keeping, or to adulteration. For this purpose the balls are heated to redness in a crucible until they no longer effervesce with acids, then powdered, and again mixed with water into balls as before. If the hardening under water take place, then it may be inferred that the cement has been spoiled by keeping, or was not well prepared.

If, on the other hand, the calcination does not effect any difference in the behaviour of the cement under water, it may be inferred that the material is bad, or that it is adulterated.

Sulphate of lime, CaO,SO_3. 68. $CaO,SO_3 + 2$ aq. 86 (*gypsum*).

The foreign substances likely to be present in gypsum, when not in considerable quantity, do not lessen its applicability for most purposes. When their proportion is large, they are sufficiently indicated by the appearance.

It may happen, however, that gypsum does not absorb water and harden, or "set," as it is called. This may be owing either to imperfect calcination or to the application of too high a temperature. In the former case, a little of the substance, heated in a tube, disengages water, which condenses in drops at the upper cold portion of the tube. The quantity of water may be estimated by igniting a weighed quantity, and then weighing again. Raw gypsum contains from 20 to 21 per cent. of water; calcined gypsum should not contain at the utmost more than 5 or 8 per cent.

Phosphate of lime, $3CaO,PO_5$.

The principal constituent of bone ash.

Bone ash should be perfectly soluble in hydrochloric acid; an insoluble residue indicates the presence of *clay* or *sand*. Digested with water, very little should be dissolved. The liquid should not redden litmus; should not give a precipitate with lime-water, which would indicate *vegetable ashes*, nor with oxalate of ammonia, which would indicate adulteration with *gypsum*. With acids it effervesces, but the carbonate of lime should not amount to more than 4 per cent. It may be estimated by means of the apparatus (fig. 33) for the estimation of carbonic acid in alkalimetry. The loss of weight in this experiment should not exceed 3 per cent.; when greater, there is reason to suspect adulteration with *chalk*. See Examination of manures.

Chloride of calcium, Ca Cl. 55·5.

Should be colourless, readily and perfectly soluble in water; the solution should not redden turmeric paper, nor become brown with sulphuretted hydrogen; caustic ammonia should not produce any precipitate. The salt when heated alone should not give off any volatile substance, nor when heated with lime evolve ammonia.

Fluoride of calcium, Ca Fl. 39·0 (*fluor-spar*).

The frequent natural association of quartz is, when considerable, indicated by the great degree of hardness and other exterior characters; in small proportion it is not detrimental for most of the applications of fluor-spar. It may be easily detected by heating the fluor-spar with four times its weight of sulphuric acid in a platinum retort, and passing the gas disengaged into water. When silica is present it is separated from the gas in white flocks.

Sulphuret of calcium, CaS and CaS, HS.

The former is used mostly in pharmacy, the latter as a depilatory. Both substances should dissolve in hydrochloric acid with disengagement of sulphuretted hydrogen. The former should not leave any considerable residue of sulphate of lime, nor present any great precipitation of sulphur. The latter should not contain any sulphate, and the precipitate formed with hydrochloric acid should be pure sulphur. The solution should not redden turmeric paper, indicative of *caustic lime*.

The latter substance is frequently obtained from the lime-purifiers of gas-works, and consequently contains carbonate of lime, which may be detected by adding, first, an excess of a concentrated solution of bichromate of potash, and then sulphuric acid; when by this treatment effervescence is produced, it is indicative of the presence of carbonate of lime. The quantity may be ascertained by introducing a weighed quantity of the substance into the flask *a* of the carbonic acid apparatus (fig. 33), together with two or three times as much bichromate of potash, and estimating, in the usual manner, the quantity of carbonic acid disengaged. Every 22 parts of carbonic acid correspond to 50 parts carbonate of lime.

Acetate of lime, CaO, $\bar{A}$. 79.

When pure is a white salt; that prepared with acetic acid from the distillation of wood is generally yellow or brown. This salt should be perfectly and readily soluble in water and alcohol. The dilute aqueous solution should not give any precipitate with nitrate of silver that is not soluble on the addition of more water. A precipitate with chloride of barium indicates the presence of sulphates, and a brown colour with sulphuretted hydrogen the presence of heavy metals—lead, copper, &c. A blue precipitate with ferro or ferricyanide of potassium in the presence of hydrochloric acid indicates iron.

The crude acetate is rarely free from one or all of these impurities in greater or less proportion. The amount of water in the salt is very variable; it is estimated in the usual manner, by drying the powdered substance at 212° F. and weighing.

Hypochlorite of lime, CaO, ClO.

The essential ingredient of bleaching powder, or, as it is called, "chloride of lime." The empirical formula of the substance is CaO + HO + Cl. Whatever may be its true chemical constitution, it does not contain, when saturated to the utmost with chlorine, more than 53 per cent. of the gas to 47 per cent. of hydrate of lime. But this degree of saturation is, in most instances, purposely avoided, because the presence of a small quantity of hydrate of lime prevents

it from such speedy alteration as it otherwise would undergo. This alteration is a main source of variation in the value of bleaching powder; for while that depends wholly upon the amount of chlorine capable of bleaching, its conversion into chloride of calcium—a substance valueless for the purposes to which this is applied—takes place very readily, especially when the temperature is somewhat raised, and the carbonic acid of the atmosphere likewise causes a small but constant evolution of chlorine. It is therefore evident that, with all these possible sources of difference in the value of the material, it is a matter of the greatest importance to the manufacturer, who consumes enormous quantities of it, to possess some means of ascertaining the value of any sample of bleaching powder with ease and certainty.

The various processes which have been adopted or proposed for the valuation of chlorinated lime are all based upon the fact, that when the evolution of chlorine takes place in the presence of some oxidizable substances and of water, a reaction takes place, which consists in the decomposition of water and a partition of its elements between the chlorine and the oxidizable substance. As the quantity of the latter which is oxidized is equivalent to the quantity of chlorine evolved, it is possible, by means of the equation representing the reaction, to calculate the amount of available chlorine in any sample of bleaching powder, either from the quantity of substance oxidized by a known weight of the bleaching powder, or from the quantity of bleaching powder necessary to oxidize a given weight of that substance.

As might be supposed, a great number of substances have been used for this purpose with more or less advantage. Those best calculated for this application are arsenious acid AsO_3 and protosulphate of iron FeO,SO_3. The reactions being respectively—

1. $AsO_3 + 2HO + 2Cl = AsO_5 + 2HCl.$
2. $2FeO,SO_3 + HO + Cl = Fe_2O_3\,SO_3 + HCl.$

In consequence of the ready disengagement of chlorine from the solution of bleaching powder, it is necessary to add it to the normal solution.

The normal solution of arsenious acid is prepared by dissolving 4·95 grms. of arsenious acid by the aid of heat in

dilute hydrochloric acid, and making up the volume with water to a litre. The normal solution of protosulphate of iron should always be made fresh, at the time it is required, by dissolving 2·78 grms. of pure crystals in water. A cubic centimeter of either solution will then be equivalent to 0·0355 grm. of chlorine.

The solution of hypochlorite should be prepared in somewhat larger quantity than will be required for the actual experiment, so as to avoid inaccuracy which might arise from the circumstance that, in the case of such a substance, a small quantity is less likely to represent the average value. According to the equation (1.) 4·95 grms. of arsenious acid are converted into arsenic acid by 3·55 grms. of chlorine, and as the per-centage of chlorine in the bleaching powder is to be ascertained, the quantity used for an experiment must be regulated according to this proportion. 35·5 grms. of the bleaching powder are mixed with water to the volume of one litre, and the whole allowed to stand until the solution becomes clear. Each cubic centimeter of this solution will represent 0·0355 grm. of the material, and the number of cubic centimeters of normal arsenious acid solution required to complete the reaction, with 100 cubic centimeters of hypochlorite solution, would give the per-centage of chlorine in the sample of bleaching powder.

As there is always a loss of chlorine when, as in other volumetrical experiments, the normal solution is added to that of hypochlorite, it is necessary to avoid the error which would thus be involved, by reversing the operation, and adding the hypochlorite solution to the solution of arsenious acid. The result is then obtained in a somewhat different manner. If, for instance, 40 cubic centimeters of hypochlorite solution are required for the oxidation of the 0·495 grm. of arsenious acid contained in 100 cubic centimeters of normal solution, the 40 cubic centimeters of hypochlorite solution will contain 0·355 grm. of chlorine, and as the 40 cubic centimeters represent $40 \times 0{\cdot}0355 = 1{\cdot}420$ grm. of the bleaching powder, the per-centage of chlorine x will be found by the proportion—

$$x = \frac{100 \times 0{\cdot}355}{1{\cdot}420} = 25.$$

The result may likewise be obtained by dividing 1000 by

the number of cubic centimeters of hypochlorite solution required to oxidize the arsenious acid in 100 cubic centimeters of normal solution. Thus, in the above instance, $\frac{1000}{40} = 25$, and if 50 cubic centimeters were required, then the per-centage of chlorine would be $\frac{1000}{50} = 20$.

When the bleaching powder to be examined is either very strong or very weak, a fraction or multiple of the above quantity may be taken in making the hypochlorite solution, and the result multiplied or divided as the case may be.

Experiment.—100 cubic centimeters of normal solution are measured into a beaker and mixed with a few drops of indigo solution, which serves to indicate the end of the reaction, for in the presence of arsenious acid it is not decolorized by chlorine, although this takes place immediately when the whole of the arsenious acid is oxidized.

The beaker containing the coloured solution is then placed upon a sheet of white paper, and the hypochlorite solution added from a burette until the blue colour suddenly disappears.

When protosulphate of iron is used as a reagent the same course is pursued, except that the end of the experiment is ascertained by testing the liquid with a solution of ferricyanide of potassium. Several drops of this reagent are placed on a white porcelain slab, and from time to time a drop of the liquid is taken out of the beaker by a glass rod and added to one of them. So long as any protosalt of iron remains a blue colour is produced, but as soon as the oxidation, is complete only a greenish-brown colour. All those methods in which it is necessary to ascertain the progress of the reaction by testing a drop of the liquid under examination are objectionable, because in this way a source of error is introduced that it is difficult to make allowance for.

A very convenient modification of the arsenious acid process for estimating the value of hypochlorites is to use arsenite of soda as the reagent.

The normal solution is prepared by dissolving 4·95 grms. arsenious acid with 14·5 grms. of pure carbonate of soda, and about 800 grms. of water, afterwards making up the volume

to one litre. 35·5 grms. of the hypochlorite are treated with water, and this solution likewise made up to one litre. 100 cubic centimeters of each solution are then measured off, and the hypochlorite gradually added to the normal solution of arsenite.

The perfect oxidation of the arsenious acid is ascertained by means of a test-paper saturated with iodide of sodium and starch. After each addition of hypochlorite the solution is stirred with a glass rod and a drop allowed to fall upon the test-paper, which remains unchanged in colour so long as there is no excess of hypochlorite, but a single drop more than is sufficient to oxidize the arsenious acid is sufficient to produce a blue colour in the paper.

The iodide of sodium and starch paper is prepared by dissolving 1 grm. of iodine, 7 grms. of crystallized carbonate of soda, and 3 grms. of potato starch in about half a pint of water, and then making up the volume to half a litre. Strips of white paper are saturated in this solution, dried and kept for use. 2 grms. of iodide of potassium may be substituted for the iodine and carbonate of soda.

The calculation of the results is made precisely in the same manner as described above.

The distinctive character of soluble calcium compounds is the formation of a white precipitate $CaO,\overline{O}$ with oxalate of ammonia in the presence of free ammonia, even when the solutions are very dilute.

Magnesium compounds.

Magnesia, MgO. 20.

Used in pharmacy and as a polishing powder, &c. It should not effervesce with acids nor yield any saline substance to water. When moistened it should not become very hot, and the neutral solution in hydrochloric acid, somewhat diluted, should not give any precipitate with oxalate of ammonia in the presence of chloride of ammonium, which would indicate the presence of lime. It should be perfectly soluble in dilute sulphuric acid, and the solution should not become turbid or brown with sulphuretted hydrogen.

Carbonate of magnesia, $3(MgO,CO_2) + MgO,HO + 3$ aq.

Used in medicine and as tooth-powder, &c., as well as for

mixing with pigments and for various chemical purposes. With the exception of effervescing with acids, it should present the same chemical characters as magnesia.

Sulphate of magnesia, $MgO, SO_3 + 7$ aq. 123.

Besides the medicinal uses of this salt, it serves as the source of other magnesian preparations. It should be white and perfectly soluble in water. A rather dilute cold solution should not give any precipitate with bicarbonate of potash, but with neutral carbonate it forms a thick pasty precipitate. When mixed with carbon and heated upon charcoal in the blowpipe flame, the remaining mass should not evolve sulphuretted hydrogen (indicative of an admixture of sulphate of soda) when moistened with an acid.

Sulphate of soda is the most frequent impurity, and it is sometimes substituted for sulphate of magnesia. It may be detected by dissolving a weighed quantity of the salt, and adding baryta-water or sulphuret of barium so long as a precipitate is formed, then heating the mixture and filtering. If the filtrate contains baryta, it must be precipitated by carbonate of ammonia and again filtered. The clear liquid evaporated to dryness should not leave any residue when the sulphate of magnesia is free from sulphate of soda.

The distinctive characteristic of magnesium compounds is the production of a pale red colour when moistened with nitrate of cobalt and heated on charcoal with the aid of the blowpipe.

Aluminium compounds.

Emery. A massive variety of corundum, occurring commercially in fine powder, used for grinding and polishing. Many natural and artificial substances are sold as substitutes for emery, either confessedly so or under its name. Among these are powdered slags, iron ores, &c. While true emery contains but little iron, from 3 to 4 per cent., and scarcely any silica, considerable quantities of these substances together with some lime and alkalies are contained in these factitious mixtures. Alumina is almost insoluble in hydrochloric acid, while the substances fraudulently substituted for it soon dissolve more or less in this acid. The nature of the admixtures may be ascertained by treating the solution according to the system described in Chap. X., and if it is desirable

to estimate the various ingredients, the necessary instructions will be found in Chap. IX.

Sulphate of alumina is sometimes brought into the market. It is a very variable substance, containing, among other impurities, iron, zinc, and lime.

A solution of the salt should not give any precipitate with tincture of galls, with ferro- or ferricyanide of potassium, which would indicate the presence of iron. Oxalate of ammonia should not give a precipitate. When an excess of caustic potash is added, and the solution heated until it is tolerably clear, sulphuretted hydrogen ought not to give a white precipitate, which would indicate the presence of zinc.

The per-centage of water in the commercial salt varies from 46 to 57 per cent. It is estimated by heating to redness a weighed quantity.

Sulphate of alumina and potash. $Al_2O_3,3SO_3+KO,SO_3+24$ aq. 502·0.

Alum. There are three kinds of true alum—potash alum, soda alum, and ammonia alum. The first and last of these are far more frequent than the soda alum. They crystallize in octohedrons. Cubical alum contains a larger proportion of alumina than the octohedral. Roman alum is of this kind.

Ammonia alum, when heated with lime, gives off ammoniacal vapour, recognizable by its odour and by the brown colour it produces in moistened turmeric paper. *Soda alum* is distinguished from potash alum by its tendency to effloresce and by its greater solubility. Its solution gives a precipitate with antimoniate of potash.

Impurities, among which *oxide of iron* for many of the purposes to which these salts are applied is the most prejudicial, may be detected in the same way as in sulphate of alumina. The quantity of iron in Roman alum is inconsiderable, and the adherent red oxide of iron is innocuous, as it is not soluble in water.

The alums do not lose the whole of their water at 212° F. The per-centage of water in soda or potash alum is indicated by the loss of weight on ignition. The water in ammonia alum cannot be estimated in this way, because it decomposes when heated, and sulphate of ammonia is volatilized.

The per-centage of alumina may be estimated by treating

the solution of a known weight of the alum with carbonate of ammonia until nothing more is precipitated. The alumina is then collected on a filter, well washed, dried, ignited, and weighed.

Acetate of alumina. As used for dyeing and calico printing, it is prepared by treating alum with acetate of lime or soda; more frequently with acetate of lead. It is very variable in composition. The differences are owing partly to the kind and partly to the quantity of substance used for decomposing the alum.

The impurities which are most injurious for the purposes to which the aluminous mordants are applied, are *copper* and *iron*. The former may be detected by immersing in the liquid a clean iron rod, upon which the copper is deposited. Iron may be detected as in alum.

Aluminous mordant is sometimes mixed with substances which will prevent it from drying quickly upon the fabric; these are *chlorides of zinc, ammonium*, or *sodium*. The presence of one or other of these substances is indicated when nitrate of silver gives a white precipitate soluble in ammonia. Zinc is detected as in sulphate of alumina; chloride of sodium by precipitating with an excess of carbonate of ammonia, filtering, evaporating the filtrate to dryness, and driving off the ammoniacal salt by heat. When chloride of sodium is present, there is a residue, the solution of which gives a precipitate with antimoniate of potash. When the mordant gives a precipitate with nitrate of silver and no indication of chloride of sodium, it contains chloride of ammonium. The detection of ammonia alone does not indicate the presence of chloride of ammonium, because the alums of commerce frequently contain ammonia in the place either of potash or soda.

When acetate of lead has been used for preparing acetate of alumina, it gives a slight brown precipitate with sulphuretted hydrogen.

When acetate of lime has been used, it gives a precipitate with oxalate of ammonia.

When acetate of soda has been used, the acetate of alumina should be free from either lead or lime. When it contains soda, without giving a precipitate with nitrate of silver, either

there is an admixture of acetate of soda or of carbonate of soda,—which may be mixed with acetate of alumina in a certain proportion without causing a precipitate,—or soda alum has been used for preparing it. In both the former cases the proportion of soda to alumina is much greater than in the latter case. In order to ascertain which of these circumstances the presence of soda is owing to, a weighed quantity of the acetate is treated with a slight excess of carbonate of ammonia, the filtrate and washings from the alumina, mixed with enough sulphuric acid to displace the acetic acid, evaporated to dryness, ignited, and weighed. When the proportion by weight of the ignited residue to the alumina is greater than 9 : 5, acetate of soda has been used. Acetate of alumina, prepared with acetate of soda, contains the sulphate of soda resulting from the decomposition, and cannot on that account be thickened with starch.

The composition of acetate of alumina depends still more upon the proportion of acetate used for decomposing the alum. Most frequently acetate of lead is used for this purpose, and it is important to know how much has been used for a certain quantity of alum. This is ascertained as follows:

About a pound of aluminous liquor is treated with an excess of carbonate of ammonia, the alumina collected on a filter, washed, ignited, and weighed. The quantity of alum x', represented by a pound of the liquor, is calculated from the result x obtained. All three kinds of alum contain nearly 11 per cent. of alumina, therefore the proportion

$$x : x' = 11 : 100$$

may be used in all instances.

The filtrate is treated with nitric acid to decompose the excess of carbonate of ammonia, and the sulphuric acid estimated by the volumetrical method with the normal solution of nitrate of lead, as described at p. 79.

The distinctive character of aluminium compounds is the production of a blue colour when moistened with nitrate of cobalt and heated with the aid of the blowpipe.

CHAPTER VII.

COMPOUNDS OF THE HEAVY METALS: THEIR CHARACTERS, THE TESTS OF THEIR PURITY, AND METHODS OF ESTIMATION.

Chromium compounds.

Oxide of chromium, Cr_2O_3. 77·4.

Known in commerce under the name of chrome-green, as a pigment for oil and enamel painting.

It is not easily soluble in acids, and may contain accidental admixtures of soluble salts, *alkaline chlorides*, *sulphurets*, and *sulphates*, but is not likely to be adulterated. A mixture of chromate of lead, prussian blue, and heavy spar is not unfrequently sold as chrome-green. *Chrome-yellow*, *carbonate of copper*, or *chalk* may be easily detected by treatment with hydrochloric acid. Oxide of chromium should, when melted with its weight of nitrate of potash, yield a yellow mass, perfectly soluble in water.

Chloride of chromium, Cr_2Cl_3 9HO. 240·0.

Recently employed as a pigment. When heated strongly a residue of oxide is left. It behaves in other respects like the oxide.

Sulphate of chromium, Cr_2O_3, $3SO_3$. 173·4.

Used in the preparation of green ink. It may be tested like other compounds of chromium, and the ink made with it may be distinguished from green ink made with acetate of copper by the addition of ammonia in excess, which gives the characteristic blue solution in the latter case, and in the former a greyish green precipitate of hydrated oxide of chromium.

Chromate of potash, KO,CrO_3. 97·9.

Opaque yellow crystals, perfectly soluble in two parts of water at 60° F., insoluble in alcohol.

Bichromate of potash, KO,2CrO$_3$. 148·6.

Deep red crystals, perfectly soluble in ten parts of water at 60° F., insoluble in alcohol. The probable impurities in this and the previous salt are,—

Sulphate of potash, indicated by a precipitate with chloride of barium in the presence of free nitric acid. The amount x may be calculated from the quantity of sulphate of baryta a found:—

$$x : a = KO,SO_3 : BaO,SO_3 = 87·2 : 116·5$$

Chloride of potassium, indicated by a precipitate with nitrate of silver in the presence of free nitric acid, and may be estimated from the (Ag Cl) found.

Nitrate of potash may be detected by distilling the salt with sulphuric acid, and testing the distillate for nitric acid.

Alumina may be detected by reducing the acid to the state of oxide by boiling with alcohol and hydrochloric acid, then boiling the green liquid with an excess of caustic soda, and testing for alumina in the filtrate with chloride of ammonium.

Chromates of lead, PbO,CrO$_3$, 122·4, and 2PbO,CrO$_3$. 274·1.

The former salt is pale yellow, the latter red. The pure salts are perfectly soluble in nitric acid. The substances met with in commerce under the names of chrome-yellow and chrome-red consist essentially of these salts in various proportions, and sometimes mixed with a variety of other substances, the most usual being—

Sulphates of lime, lead, baryta—remain undissolved by nitric acid (the two former for the most part). The residue may be examined further, as directed in Chapter X.

Carbonate of lime is indicated by effervescence with acids, and the lime may be tested for after reducing the chromic acid and removing the oxide from solution.

Chromate of zinc, ZnO,CrO$_3$. 91·3.

A yellow powder, insoluble in water, liable to contain the same admixtures as the lead salts, and may be tested in a similar manner.

The distinctive character of chromium (as a base) compounds in solution is the formation with caustic potash of a bluish-green precipitate, Cr_2O_3, HO, soluble in an excess of

the reagent, forming a green liquid, from which the oxide may be again precipitated by boiling with chloride of ammonium.

The red or yellow colour of all chromates is to some extent indicative of chromium existing in the state of acid, and the change to green by boiling with hydrochloric acid and alcohol, furnishes a satisfactory confirmation.

The estimation of chromium, when in the state of chromic acid, may be effected very easily, in virtue of the readiness with which it gives up oxygen to some other substances. The reaction with protochloride of iron in the presence of hydrochloric acid,

$$6FeCl + 2CrO_3 + 6HCl = 3Fe_2Cl_3 + Cr_2Cl_3 + 6HO,$$

is well suited to this purpose.

The amount of chromic acid x is calculated from the quantity of iron F converted into perchloride, and this is found by estimating, as described at p. 126, how much of a known quantity of iron used remains in the state of protochloride; then

$$x : F = Cr : Fe_3.$$

The quantity of material to be operated upon in order to obtain direct per-centage results is according to the substance sought for.

		grm.
Chromium	Cr	0·560
Oxide of chromium	Cr_2O_3	0·800
Chromic acid	CrO_3	1·040

The experiment is made by dissolving 1·68 grm. of clean iron wire in 20 or 30 cubic centimeters of hydrochloric acid, and adding the substance in which chromium (in the state of chromic acid) is to be estimated. The liquid immediately acquires a deep green colour and, when a sufficient excess of hydrochloric acid is present, remains clear. When too little acid is used a precipitate ($CrO_3 + Cr_2O_3$) is formed.

Insoluble substances, in which the chromium exists in the state of oxide, must be subjected to a preliminary treatment, for the purpose of converting it into the state of chromic acid.

A weighed quantity of the substance in fine powder and well dried is projected in small portions into melted hydrate

of potash contained in a silver crucible, and when the whole is well mixed, small fragments of fused chlorate of potash (KO,ClO_5) are added from time to time, so that there may be no loss from effervescence.

The mass gradually becomes yellow, and finally transparent; it is then allowed to cool, placed in a beaker with water, and, when dissolved, the crucible removed; hydrochloric or sulphuric acid is then added to the cold liquid until it becomes orange-coloured.

When hydrochloric acid is added to the liquid while hot, an error would be caused by the reduction of chromic acid.

The end of the reaction between the residual protochloride of iron and the normal solution of permanganate is not at all disguised by the green colour of the liquid.

Zinc and its compounds.

Sulphate of zinc, ZnO,SO_3HO, + 6 aq. 143·6.

The pure salt forms transparent colourless crystals, readily and perfectly soluble in two or three times their weight of water at 60° F. Of the usual impurities the most important is—

Iron, indicated on the addition of ammonia by a brown precipitate insoluble in an excess of the reagent.

Adulteration is not probable except in the insoluble compounds.

Carbonate of zinc, ZnO,CO_2. 82·6.

The native carbonate, known under the name of calamine, is the chief source of zinc and its compounds. When purified by washing, it is used in medicine, and likewise the precipitated salt $2ZnO,CO_2 + 3ZnO,HO$.

Oxide of zinc, ZnO. 40·6

Is used as a substitute for carbonate of lead as a pigment, and is sometimes adulterated with

Sulphates of baryta and lime, clay, chalk, &c. The former may be detected by their insolubility; the latter by effervescence with acids.

The distinctive character of zinc compounds in solution is the formation with sulphuret of ammonium of a white gelatinous precipitate ZnS, insoluble in excess of the reagent and in potash.

In the estimation of zinc it is always weighed in the form of oxide, which is obtained by igniting the precipitate of carbonate or sulphuret; but, as there is a liability to error by this method, unless greater precautions are taken than would be consistent with the object of the experiment in most instances, it is better to have recourse to the volumetrical method, based upon the reaction between sulphuret of zinc and perchloride of iron:

$$ZnS + Fe_2Cl_3 = ZnCl + 2FeCl + S.$$

Any of the compounds, ores, or alloys of zinc may be examined by this method. The zinc is converted into the state of sulphuret, and the direct experimental result is the quantity of iron f reduced to the state of protochloride, while the quantity of zinc x which it represents is found by the proportion—

$$x : f = Zn : Fe_2.$$

When the quantity of substance q represented by the sulphuret of zinc operated upon is known, the per-centage x' of zinc, or of its compound sought, may be calculated; for

$$x' : 100 = x : q.$$

When $q = 1{\cdot}6$ grm. and the solution of permanganate is of the usual normal value (p. 123), the number of cubic centimeters requisite to perchloridize the iron reduced by the sulphuret of zinc gives at once the per-centage of zinc in the substance examined.

Experiment. The zinc is precipitated from the solution of the weighed substance as sulphuret, by sulphuret of ammonium, after the separation of any substances that would interfere with the result. The whole is allowed to stand in a covered beaker until the precipitate has perfectly subsided, the liquid decanted off, and the sulphuret collected upon a filter, moistened with boiling water, well washed with water —containing sulphuret of ammonium to prevent oxidation— then transferred, together with the filter, to a flask containing a dilute solution of perchloride of iron with a slight excess of acid, and gently warmed until the sulphur aggregates at the bottom. The amount of protochloride of iron is then estimated with permanganate solution in the usual manner.

When the substance in which zinc is to be estimated contains copper or other metals precipitable by sulphuretted

hydrogen, they must be separated by treating the solution with that gas after adding hydrochloric acid. Iron and manganese may be separated from the filtrate or from the original solution, after boiling with a few drops of nitric acid to convert them into the state of peroxide, by an excess of ammonia, in which the oxide of zinc is soluble, and from which it may be precipitated as sulphuret by sulphuretted hydrogen.

In calamine containing carbonate and silicate of zinc, the former may be estimated by igniting a weighed quantity and digesting it with ammonia and carbonate of ammonia, which dissolves the carbonate of zinc alone.

When the zinc has to be separated from other metals before conversion into sulphuret, it will be advisable to take a multiple quantity of the substance and to operate upon a corresponding fraction of the ammoniacal solution or of that containing the protochloride of iron.

Manganese compounds.

Binoxide of manganese, MnO_2. 43·6.

The native binoxide, called *pyrolusite* by mineralogists, and known in commerce under the name of manganese, is extensively used as a means of generating chlorine for the preparation of bleaching powder, &c. It is rarely pure, and contains variable proportions of other minerals. Its commercial value depends upon the quantity of chlorine which a given quantity of it is capable of liberating, when heated with hydrochloric acid, in consequence of the reaction represented by the equation

$$\overbrace{MnO + O} + 2HCl = MnCl + 2HO + Cl,$$

according to which 4·36 grms. of binoxide of manganese liberate 3·54 grms. of chlorine, and $\frac{1}{100}$ of that quantity 0·035 grm. chlorine.

The per-centage of MnO_2, or of available oxygen in any sample of manganese, may therefore be ascertained by estimating the quantity of chlorine generated by 4·36 grms.

A great number of methods, both volumetrical and gravimetrical, have been proposed for the valuation of manganese, and of each class some one or more have come into general use as the most convenient.

I. *Volumetrical methods.*

The reaction that takes place between nascent chlorine and oxidizable substances may be applied to the valuation of manganese as well as of bleaching powder (p. 97).

For this purpose the chlorine generated by heating a mixture of the manganese with excess of HCl, is dissolved in a solution of caustic potash, and its quantity estimated by means of the normal solution of arsenious acid precisely as in the valuation of bleaching powder.

Experiment. The former part of the operation is most conveniently conducted in the following manner. An average sample of the manganese is reduced to powder, and 4·36 grms. introduced into the small flask A, about 5 centimeters in diameter.

Fig. 39.

A long bent tube is fitted to this flask by means of a tight cork, and inserted into a long-necked flask B, of about half a litre capacity, filled to the bottom of the neck with a weak solution of caustic potash, and placed in an inclined position as shown in the figure. The peroxide of manganese is weighed upon a piece of paper, in which it is wrapped after the fashion of a cartridge, and dropped into the flask A, containing 25 cubic centimeters of concentrated hydrochloric acid. The cork is then rapidly replaced, and heat applied gradually. The chlorine disengaged drives the air contained in A, into the upper part *b* of the flask B, and makes the liquid rise up the neck, so that the gas has to pass it twice before it can escape. Towards the end of the operation, the liquid in A is made to boil, and the steam carries over the whole of the chlorine into B. When the evolution of chlorine has perfectly ceased, the cork is taken out, the delivery tube rinsed with water, the volume of the liquid made up to

one litre, and an experiment made with 100 cubic centimeters, added to the normal solution of arsenious acid, precisely as in the valuation of bleaching powder.

Another method is based upon the conversion of protochloride of iron into perchloride by means of binoxide of manganese according to the equation

$$MnO_2 + 2FeCl + 2HCl = MnCl + Fe_2Cl_3 + 2HO.$$

Thus 4·36 grms. of binoxide convert into perchloride 5·60 grms. of iron existing as protochloride, by generating 3·54 grms. of chlorine; so that, by operating with this quantity of iron $= f$, and subsequently estimating how much $= f'$ remains in the state of protochloride, the amount of binoxide x, in a sample of manganese, may be ascertained from the quantity of iron F converted into perchloride by the proportion

$$x : 4{\cdot}36 = F\ (=f-f') : 56.$$

In the case of pure binoxide f' would be $= 0$.

The quantity of iron f' remaining in the state of protochloride is estimated by means of a solution of chlorate of potash, and as in this reaction

$$KO,ClO_5 + 12FeCl + 6HCl = KCl + 6Fe_2Cl_3 + 6HO,$$

2·043 grms. of chlorate are equivalent to 3·54 grms. chlorine, that quantity dissolved in water to the volume of 100 cubic centimeters will give a normal solution of which each cubic centimeter is equivalent to 0·044 grm. of MnO_2 or 0·056 grm. Fe, and consequently, the number obtained by deducting from 100, the volume of this solution ($=n$ cubic centimeters) requisite for converting into perchloride, the iron remaining as protochloride, will give the per-centage of binoxide of manganese in the sample examined.

Fig. 40.

The experiment is made with a glass flask (fig. 40) of about 400 cubic centimeters capacity, fitted with a good cork through which passes a long funnel tube *a*. 5·628 grms. of clean iron wire are introduced into the flask with about 25 cubic centimeters of pure hydrochloric acid, the cork placed loosely in the mouth, so that the end of the tube may just touch the surface of the

liquid, and heat applied until the iron is dissolved. The weighed quantity of manganese, wrapped in a piece of paper, is then dropped into the flask and the cork pressed tightly in. The chlorine disengaged is absorbed by the perchloride of iron, and any escape, even in consequence of rapid evolution, is prevented by the tube *a*, in which the liquid rises.

When the liquid falls in the tube, and the chlorine is perfectly absorbed, the cork is loosened, a strip of paper, saturated with solution of indigo, slipped between it and the neck of the flask, the cork again pressed in tight, and the normal solution of chlorate added from a burette through the funnel tube, until the colour of the paper suddenly disappears. The volume is then read off, half a cubic centimeter deducted, as a correction for the chlorine consumed in bleaching the indigo paper, and the calculation made as above directed.

Gravimetrical processes.

a. When oxalic acid is treated with sulphuric acid, in the presence of peroxide of manganese, a quantity of it, equivalent to the oxygen liberated, is converted into carbonic acid which is disengaged.

$$C_2O_3 + MnO_2 + SO_3 = 2CO_2 + MnO,SO_3.$$

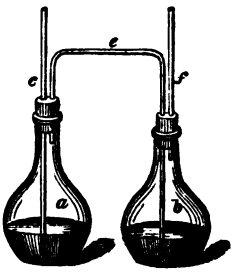
This reaction may be applied by conducting the carbonic acid into baryta water, and calculating the per-centage of oxygen, or of binoxide in the manganese from the weight of carbonate of baryta obtained.

It is also possible to estimate the value of manganese, by aid of the same reaction, by calculation from the weight of carbonic acid disengaged, when the experiment is conducted in the flask apparatus (fig. 38).

Thus by substituting for the symbols in the above equation their numerical values, we find that 4·36 peroxide of manganese represent 4·4 carbonic acid. Consequently 1·0 grm. of carbonic acid represents 0·99 peroxide of manganese; but as it is desirable not to have less than 1·0 grm. of carbonic acid to estimate, the quantity of manganese to be taken for this experiment is $0{\cdot}99 \times 3 = 2{\cdot}97$, and then the weight of carbonic acid found, divided by 3 and expressed in centigrammes, gives the per-centage of peroxide in the commercial substance.

The experiment is made by introducing the powdered manganese into the flask *a*, with 8 grms. of oxalate of potash ($\dot{K}O,\bar{O}$) and water to one-third its capacity. It is then connected by means of the tube and cork with the flask *b*, containing sulphuric acid, and the whole weighed. The tube *c* is then closed, and some of the acid transferred to *a* by sucking out air at *f*. When the reaction has subsided, a second portion of acid is transferred to *a*, and this is repeated until carbonic acid is no longer generated, and no particles of manganese remain undecomposed. When it is ascertained that there is an excess of sulphuric acid in *a*, the cork is removed from *c*, carbonic acid completely displaced from both the flasks, by drawing air through them, and the apparatus again weighed. The loss of weight represents carbonic acid, and the result is calculated as above.

Fig. 41.



When, as is not unfrequently the case, manganese contains an admixture of carbonates, the oxalic process would give a deceptive result, if this source of error were not removed, for their carbonic acid would be disengaged together with that generated from the oxalic acid. The presence of carbonates in the manganese is easily detected by the effervescence which occurs when a fragment is moistened with an acid.

In order, therefore, to remove this source of error, in the valuation of manganese containing carbonates, the weighed quantity of substance must be treated in the flask *a* with an excess of dilute nitric acid. The clear liquid is drawn off with a siphon, the manganese washed several times with water, and the valuation experiment then made as above.

b. Sulphurous acid process. When chlorine is brought in contact with a solution of sulphurous acid, an equivalent quantity of sulphuric acid is produced.

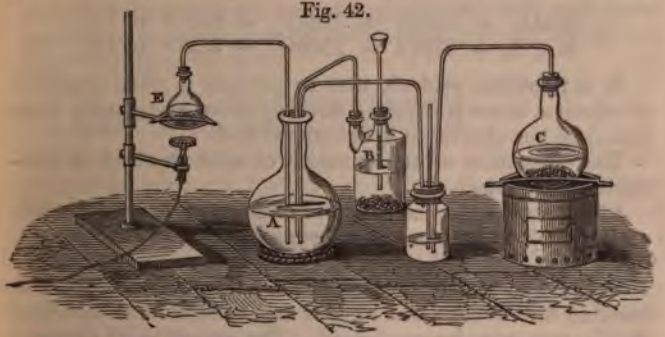
$$SO_2 + Cl + HO = SO_3 + HCl.$$

A solution of sulphurous acid, when kept for any time, always becomes more or less oxidized, and as the accuracy of the result furnished by this process depends upon the

absence of sulphuric acid, it is advisable to conduct the operation in the following manner.

The flask A is half-filled with water freed from air by boiling, and a certain quantity of chloride of barium added. A current of hydrogen, generated in the vessel B, is then passed through the water until it becomes cold.

Fig. 42.



The air having been displaced from the flask A, sulphurous acid is generated by heating a mixture of sulphuric acid and copper in the flask C. This gas is washed in the small bottle D, containing water. Lastly, the weighed quantity of manganese is rapidly introduced into the flask E, containing hydrochloric acid. The chlorine here generated passes into A, where an equivalent quantity of sulphuric acid is formed. When the whole of the chlorine has passed over, the liquid in A is boiled to expel the excess of sulphurous acid, oxidation of which meanwhile is prevented by keeping up the current of hydrogen. The sulphate of baryta is then collected on a filter, weighed, and the result calculated according to the above equation.

The binoxide of manganese (pyrolusite) is sometimes accompanied by other oxides of manganese, or by substances which consume hydrochloric acid without generating chlorine. In order therefore to estimate completely the value of a sample of manganese, it is not alone sufficient to ascertain the quantity of chlorine it is capable of liberating, but the quantity of acid consumed must likewise be taken into account.

The substances which are likely to be present in manganese and cause a larger consumption of acid, are earthy carbonates, oxide of iron, and likewise some other oxides of manganese. The former act simply by neutralizing a portion of the acid, and the sesquioxide of manganese has to a certain extent the same influence. Thus:—

$$MnO_2 + 2HCl = MnCl + 2HO + Cl.$$
$$Mn_2O_3 + 3HCl = 2MnCl + 3HO + Cl.$$

It is therefore evident, that to produce a given quantity of chlorine, there is required not only more sesquioxide than binoxide, but also more hydrochloric acid.

The estimation of the quantity of acid required is conducted as follows:—

4·36 grms. of the manganese, in fine powder, are mixed in a glass flask with 40 grms. of hydrochloric acid (1·09 sp. gr. or 18·2 per cent. HCl), and one end of a bent tube fitted by means of a cork into the neck of the flask, while the other is inserted into a wet flask, so that any hydrochloric acid that distils over may be condensed. The evolution of chlorine is then facilitated by heat, and when at an end, the liquid is diluted and the quantity of acid remaining ascertained, either by means of a normal solution of alkali, or by the following method.

Some pieces of marble, of known weight, are dropped into the liquid, and when, after the application of a gentle heat, there is no further evolution of carbonic acid, the pieces of marble are taken out, washed, dried, and weighed. The quantity of acid taken for the experiment, less that neutralized by the marble, or indicated by the volumetrical experiment, gives the quantity consumed in generating chlorine.

The quantity for 4·36 grms. of substance would be 7·30 grms. for MnO_2, and 6·00 grms. Mn_2O_3.

If in an experiment the quantity of hydrochloric acid taken represents 7·30 grms. HCl, and the remainder is estimated at 0·73 grm., the quantity consumed in generating chlorine will be 7·30 − 0·73 = 6·57.

Supposing that in a previous experiment 4·36 grms. of the manganese have been found to generate with hydrochloric acid 2·6 grms. of chlorine, then as the HCl consumed in generating chlorine by means of MnO_2 should bear the same

proportion to the quantity of chlorine liberated, as 73 : 35·5, the acid x, consumed in generating 2·6 grms. of chlorine, should be

$$x : 2{\cdot}6 = 73 : 35{\cdot}5 \quad (x = 5{\cdot}34 \text{ grms.}).$$

But in the above experiment it was found to be 6·57 grms., consequently 1·23 grm. were consumed by impurities, and for generating a given quantity of chlorine with such manganese, 25 per cent. more hydrochloric acid would be required than with pure binoxide.

Iron and its compounds.

Sulphate of iron, FeO, So_3 + 7HO. 139.

Transparent crystals of a uniform pale green colour, without white or brown spots or incrustation, perfectly soluble in water, and when dried at 212° F. loses 44 per cent. of water. The probable impurities are,—

Copper, indicated by the blue colour of the liquid remaining after the addition of ammonia, in excess, to the solution; and by a red metallic coating upon a piece of bright iron dipped into the solution.

Persalts of iron, indicated, on the addition of ammonia, by a brown or dark green colour of the precipitate, which should be dirty white.

Zinc, indicated by the formation of a white precipitate on evaporating the filtrate after precipitation with ammonia.

Alumina, and also *zinc*, are indicated, when the solution is mixed with excess of caustic soda, the filtrate neutralized with acid and treated with carbonate of soda, by a white gelatinous precipitate.

Persulphate of iron, Fe_2O_3, $3SO_3$. 200.

The dry, neutral salt is white, dissolves in water, slowly but completely, with a reddish-brown colour. Ferricyanide of potassium should not give a blue colour with it.

As this salt is mostly prepared from protosulphate, it may contain the same impurities, and is to be tested in the same manner.

Nitric acid is indicated by the decoloration of a drop of indigo solution when heated with a solution of the salt.

Pernitrate of iron, Fe_2O_3, $3NO_5$. 240.

Occurs in commerce in solution. It should not contain

any considerable excess of *nitric acid*; this may be ascertained by digesting the solution with clean iron filings in a warm place; no evolution of red nitrous vapour should appear, and when the dilute solution is boiled, a basic salt should be precipitated.

Sulphuric acid should be tested for, because sulphate of iron, treated with nitric acid, is sometimes fraudulently substituted for this salt.

Acetates of iron, $FeO,\bar{A}$. 87, and $Fe_2O_3\bar{3}\bar{A}$. 233.

For most purposes the protacetate is required, but peracetate is always present. The proportion of each may be ascertained by one of the processes described at p. 125.

Protochloride, FeCl. 63·5, and Perchloride of iron, Fe_2Cl_3. 162·5.

The former salt should not give a blue precipitate with ferrocyanide of potassium, indicative of persalt.

The latter should not contain nitric acid, or give a blue precipitate with ferricyanide of potassium.

Ferrocyanide of potassium, K_2Cfy. 184·4. $K_2Cfy + 3HO$. 211·4.

Yellow semitransparent crystals. The probable impurities are,—

Alkaline carbonates, indicated by effervescence when a crystal is dipped into hydrochloric acid.

Sulphates, indicated by a precipitate with chloride of barium in the presence of free acid.

Alkaline chlorides, in small proportion, are generally present in ferrocyanide of potassium, and must be considered as an adulteration only when the quantity is large. They may be detected and, if required, estimated by treating a known quantity of the substance with two or three times as much nitrate of potash and ten times as much carbonate of soda, igniting the mixture, and testing the filtered solution, to which a slight excess of nitric acid has been added, with nitrate of silver.

Valuation of ferrocyanide of potassium.

The reaction between this compound and persalts of iron may be applied to this purpose.

$$3(K_2Cfy) + 2(Fe_2O_3,3SO_3) = Fe_4Cfy_3 + 6KO,SO_3.$$

The test solution is prepared by treating a hot solution of

87·67 grms. of pure crystallized sulphate of iron with nitric acid, added in small quantities until red vapours are no longer generated and the liquid ceases to give a blue colour with ferricyanide of potassium. It is then diluted with water to the volume of one litre. 100 cubic centimeters of this solution are equivalent to 10 grms. of ferrocyanide.

The test solution may likewise be prepared by estimating the quantity of iron in 100 cubic centimeters of a moderately dilute solution of a persalt of iron, and thence calculating the quantity of substance to be taken for an experiment as the equivalent weight of ferrocyanide.

The quantity of material for an experiment is 10 grms., which is dissolved in water and slightly acidulated with hydrochloric acid. A correction of the results obtained by this process is necessary, on account of the mechanical abstraction of a portion of the ferrocyanide from solution by the precipitate formed. This, under the conditions of the experiment, amounts to about $\frac{1}{10}$th of the ferrocyanide present, so that if with 10 grms. of substance, the reaction is completed by 60 cubic centimeters of test solution, it would contain 63 instead of 60 per cent. of ferrocyanide of potassium.

The experiment is made in the usual manner with a burette, but as the precipitate of prussian blue remains for a long time suspended in the liquid, communicating to it an intense blue colour, a special contrivance for ascertaining the end of the reaction is necessary. This consists in allowing a drop of the liquid, coloured by the suspended precipitate, to fall upon a piece of white bibulous paper, and touching the moist ring, which is formed round the spot where the drop fell, with a glass rod moistened with an appropriate reagent. A blue colour will then be produced, before the end of the reaction, by a persalt of iron, and after it by ferrocyanide of potassium.

It must be remembered,—1st, that the colour is not produced at the outer edge of the moist ring, but more towards the centre; and, 2nd, that it does not become visible until after a few moments.

The presence of sulphocyanides does not invalidate the result, because sulphocyanide of iron is not precipitated until all the ferrocyanide is removed from solution.

Ferricyanide of potassium, K_3Cfy_2. 329·6 (*red prussiate*).

The pure salt forms transparent, columnar crystals of a ruby colour, soluble in 3·8 parts of water and in less alcohol.

The solution of this salt, and the pulverulent material sold for dyeing purposes, are more liable to contain admixtures, and require valuation on account of the possible variation in the amount of red prussiate they contain.

As ferricyanide of potassium may be converted into ferrocyanide by reducing agents, the process already described for the estimation of ferrocyanide may likewise be applied to this salt.

For this purpose the substance in solution is boiled with a few grammes of sulphite of soda and a little caustic potash. Generally there is not any precipitate, but when there is, it must be separated by filtration. The solution of ferrocyanide thus obtained is then acidified, the normal solution of persalt of iron added from the volumeter, and the experiment conducted in the manner already described, p. 119.

The quantity of material to be operated upon in order to obtain a result expressing the per-centage of red prussiate, is 7·99 grms., or some multiple of it. The amount of ferricyanide x' may in any instance be calculated from the quantity of ferrocyanide x, indicated by the volume of test solution required to complete the reaction, by means of the proportion:

$$x' : x = K_3Cfy_2 : 2K_2Cfy.$$

The reduction of ferricyanide of potassium into ferrocyanide may be effected by iron and caustic potash, but even then filtration is necessary.

The reaction between ferricyanide of potassium and sulphide of sodium,

$$K_3Cfy_2 + NaS = 2\left(\left[\begin{smallmatrix}K\\Na\end{smallmatrix}\right]_2 Cfy\right) + S,$$

would furnish a direct indication of the amount of the salt, but as sulphide of sodium is not a very stable compound, sulpharsenate of sodium ($3NaS,AsS_5+15$ aq.) has been substituted for it.

To obtain the desired reaction with this salt, it is requisite that the solution of ferricyanide should be alkaline:

$$6K_3Cfy_2 + 3NaS,AsS_5 + 3NaO = 12\left(\left[\begin{smallmatrix}K\\Na\end{smallmatrix}\right]_2 Cfy\right) + AsO_5.$$

The point at which the reaction ends is indicated by the addition of a drop of cochineal decoction, which is immediately bleached so long as a trace of ferricyanide remains in solution.

The reagent is prepared by dissolving pentasulphide of arsenic in solution of sulphide of sodium. The crystals formed on evaporation are pressed between bibulous paper, dried at 212° F., and preserved in a well-stopped bottle.

The experiment is made with 5 grms. of the red prussiate and 1 grm. of the sulpharsenate, dissolved with 3 grms. of carbonate of soda, in water to the volume of 100 cubic centimeters. Each cubic centimeter is then equivalent to 0·1 grm. of red prussiate.

At the ordinary temperature the presence of carbonate of soda does not effect the decomposition of ferricyanide with sufficient rapidity to influence the results, but it is probable that the whole method is too operose to answer the requirements of technical analysis.

The reaction between ferricyanide and protochloride of tin in the presence of hydrochloric acid may be applied to the valuation of this substance. It is:—

$$2(K_3Cfy_2) + 2HCl + 2SnCl = 3(K_2Cfy) + H_2Cfy + 2(SnCl_2).$$

The end of the reaction is indicated by the transition from the green colour, at first assumed, to a distinct violet or blue.

Ferrocyanide of iron, Fe_4Cfy_3.

This substance, in a pure state, is a compact, amorphous mass of an intense blue colour. The fracture is conchoidal, and when rubbed assumes a copper lustre. It occurs in commerce in various states of purity, and under the names of Chinese, Paris, and Prussian blue.

The substances with which it is usually mixed are alumina, oxide of zinc, clay, chalk, and gypsum. Most of them may be detected by treating the material, in fine powder, with a little hydrochloric acid, by which they are dissolved. Their further recognition may be effected in the filtered liquid by means of the usual tests. *Oxide of zinc* may be detected by digesting a portion of the material with caustic soda, and testing the filtrate with sulphide of ammonium.

Sulphate of baryta or *gypsum* may be detected, after igniting the powdered material together with a small quantity of

sugar, by treating the residue with hydrochloric acid, when sulphuretted hydrogen is evolved, and baryta or lime may be tested for in the solution.

Starch is indicated when the material is boiled with a little water, by the formation of a gelatinous mass, which again becomes liquid when boiled with a few drops of sulphuric acid.

Valuation of pigments containing prussian blue.

A number of blue pigments, occurring in commerce, consist of prussian blue mixed with a variable proportion of some white powder. Some green pigments likewise consist of prussian blue and chrome-yellow, frequently with a white powder, or a yellow inorganic substance. A simple method of determining the quantity of prussian blue in such pigments would often be serviceable, and as prussian blue may readily be converted into ferrocyanide of potassium, the above method may be applied for this purpose. The experiment is conducted as follows:—

In order that the test liquid prepared as above may be used, 6·790 grms. of the pigment to be examined is weighed, boiled with caustic potash until the blue or green colour is destroyed, the liquid filtered, the residue well washed with hot water, and the filtrate containing ferrocyanide of potassium treated in the manner already described. As in this case there is no sulphocyanide of potassium present, the addition of the test solution of iron and the testing of the liquid on paper with a persalt of iron may be continued until the reaction with the latter ceases. If it so happens that the blue precipitate does not settle readily upon the paper, but spreads out over the moist ring, this inconvenience may be prevented by the addition of some solution of chloride of sodium, or any other indifferent salt. This spreading of the colour is owing to the formation of a little prussian blue that is soluble in water, though not in a strong saline solution.

The distinctive character of iron compounds in solution is the formation with ferrocyanide or ferricyanide of potassium of blue precipitates; the former reaction being indicative of persalts, the latter of protosalts.

Volumetrical estimation of iron.

All the processes by which this is effected are based upon the conversion of protochloride of iron, FeCl, into per-

chloride, Fe_2Cl_3, and the ease with which iron in solution may, for the purpose of estimation, be reduced to the state of protochloride.

The reagents used for the estimation of iron by this method are various; with permanganate of potash the reaction is represented by the equation:—

$$10FeCl + 7HCl + Mn_2O_7 = 5Fe_2Cl_3 + 2MnCl + 7HO.$$

The normal solution is prepared by estimating the volumetrical value of a solution containing about the proper proportion by a direct experiment with pure iron.

For this purpose 1·4 grm. of clean iron wire is dissolved in a flask by 25 cubic centimeters of pure hydrochloric acid, diluted with water. It is not necessary that the water should be boiled free from air, nor is there any danger of the iron passing into the state of perchloride in the presence of the excess of acid used.

The pale green solution is diluted with thirty times its volume of water, and when quite cold, the solution of permanganate gradually added from a burette, the mixture being well agitated after each addition. As soon as the iron is converted into perchloride, a single drop of permanganate is sufficient to give to the liquid a very decided red colour, which indicates the end of the reaction. The number of cubic centimeters used is then observed, and water added to the permanganate in such proportion as to make 100 cubic centimeters represent 2·8 grms. of iron. Thus if 1·4 grm. of pure iron requires 40 cubic centimeters, the solution must be diluted with $\frac{1}{4}$ its volume of water to bring it to the normal value.

A cubic centimeter of this solution is equivalent to 0·028 grm. of iron, and the amount of iron x in a solution may be ascertained by multiplying the number of cubic centimeters C_n required for its conversion into persalts by this number,

$$x = C_n \times 0{\cdot}028.$$

When the quantity q of material represented by x is known, the per-centage of iron x' may be found by the proportion:—

$$x' : 100 = x : q.$$

If q corresponds with the equivalent of iron, or of the

compound of iron sought, the number of cubic centimeters of normal solution used expresses at once their per-centage.

Quantity of material. The numbers below indicate the quantities to be taken in order to find the per-centage of iron or the compounds sought:—

Iron	Fe	2·8.
Oxide of iron	FeO	3·6.
Peroxide of iron	Fe_2O_3	4·0.
Sulphate of iron	FeO,SO_3+7HO	13·9.

The iron solution must be *dilute* and *cold* before the permanganate is added, because otherwise an error would be caused by the evolution of chlorine, owing to a reaction represented by the equation:—

$$Mn_2O_7 + 7HCl = 5Cl + 2MnCl + 7HO.$$

Experiment. The first step is to obtain a solution of the iron in the state of protochloride.

The assay mass is introduced into a flask, and boiled with concentrated hydrochloric acid until the whole is dissolved, or the residue of silica, clay, &c. has a pure white colour. Metallic iron is weighed in the form of turnings; most ores require to be powdered. The solution is generally effected very readily, but when this is not the case a few drops of nitric acid must be added. The perchloride of iron thus formed, or arising from the presence of peroxide in the ore or other substance examined, must, of course, be reduced before adding the normal solution.

The reduction of perchloride may be best effected by heating the iron solution with metallic zinc,

$$Fe_2Cl_3 + Zn = 2FeCl + ZnCl.$$

For this purpose the zinc, which must of course be perfectly free from iron, is cast in the form of a ball upon a thin platinum wire.

The reduction may likewise be effected by boiling the iron solution with 4 or 5 grms. of crystallized sulphite of soda, but the time occupied in separating the excess of sulphurous acid renders this method inferior to the former, which, moreover, presents the additional advantage of ensuring the separation of any traces of copper or arsenic. The occurrence of these metals in iron ores is not unfrequent, and as they

resemble protosalts of iron in their behaviour with permanganic acid, their presence in the chloride solution would lead to inaccurate results.

Besides copper and arsenic, none of the other metals which usually occur in iron ore have any prejudicial influence upon the experimental results obtained by this method.

When cast iron is dissolved in hydrochloric acid, it frequently happens that some oily or carbonaceous substance is separated, and as this would reduce a certain amount of permanganate, the chloride of iron must in such cases be evaporated to dryness, ignited with chlorate of potash, redissolved in hydrochloric acid, and then reduced by zinc. Care must always be taken to prevent the introduction of any organic substance into the iron solution.

When the whole of the iron in the solution has been reduced to the state of protochloride, it is considerably diluted with water and the normal solution of permanganate added from a burette, in the same manner as described for the valuation of the latter by means of pure iron.

It may sometimes be desirable, as for instance in the case of iron ores, to ascertain the relative quantities of iron present in the state of proto- and peroxide. For this purpose two experiments must be made; one, with the simple solution of the substance in hydrochloric acid, gives the quantity of protoxide; a second, with the chloride solution, previously reduced by zinc, gives the total amount of iron, and the difference between the two, the quantity of peroxide.

The solution of permanganate decomposes gradually, and when kept for any time its value is consequently reduced, so that before using it, a preliminary experiment with iron wire must always be made, and, if necessary, valuation results corrected. Thus, if 1·4 grm. of iron require 51·3 instead of 50 cubic centimeters of solution, a corresponding deduction from the result of each experiment must be made.

The mutual convertibility of proto- and perchloride of iron by oxidizing or reducing substances is frequently applicable to purposes of valuation. Thus the amount of any reducing substance, which has a definite reaction with perchloride of iron, may be indirectly estimated according to the equation representing the reaction from the quantity of protochloride formed, this being ascertained by means of the normal solu-

tion of permanganate. Let x be the substance sought, f the iron converted by it into protochloride, A and B their proportions in the reaction, the amount of x may be found by the proportion:—

$$x : f = A : B.$$

The amount of any oxidizing substance x' which has a definite reaction with protochloride of iron may be found in a similar manner by operating with a known quantity of iron (2·8 equivalent to 100 cubic centimeters of permanganate solution) in the state of protochloride, and estimating how much iron f remains in that state after the reaction. The quantity of iron F converted into perchloride will then be:—

$$F = 2{\cdot}8 - f, \text{ and } x' : F = A : B.$$

Such a general applicability of a volumetrical reagent considerably enhances its practical value. The substances to which it may be applied, and the equations by which their amount is found, are as follows:—

Nitric acid	$x : F = NO_3$	$: Fe_6$.
Chromic acid	$x : F = CrO_3$	$: Fe_3$.
Binoxide of manganese	$x : F = MnO_2$	$: Fe$.
Zinc.....	$x : f = Zn$	$: Fe_2$.
Copper.....	$x : f = Cu$	$: Fe$.
Lead.....	$x : F = Pb$	$: Fe_3$.
Iodine	$x : F = I$	$: Fe_2$.

The reaction between protosalts of iron and chromic acid,

$$6FeCl + 6HCl + 2CrO_3 = 3Fe_2Cl_3 + Cr_2Cl_3 + 6HO,$$

may be taken advantage of for the volumetrical estimation of iron.

The normal solution is made by dissolving bichromate of potash in water, in such proportion that 100 cubic centimeters contain 1·477 grm.

The substance to be examined is dissolved, if necessary, in hydrochloric acid, the iron reduced to the state of protosalt by means of zinc, and the normal solution added from the volumeter.

The end of the reaction is ascertained by means of a solution of ferricyanide of potassium, several drops of which are placed on a white porcelain slab, and when a drop of the

liquid ceases to give a blue colour with this reagent, the whole of the iron is converted into persalt.

This process is inferior to that with permanganate.

Uranium compounds.

Oxide of uranium, U_2O_3.

This substance, in the anhydrous state, is a brick-red powder: the hydrate is yellow. It is used in enamel painting and for colouring glass.

The impure native oxide, known as pechuran or pitch-blende, is the principal source of uranium compounds.

Nitrate of uranium, $U_2O_3NO_5 + 6HO$.

This is the only salt met with in commerce; it is in pale yellow crystals, very soluble in water.

The distinctive character of uranium compounds in solution is the formation of a yellow precipitate with caustic potash.

Cobalt compounds.

The most important application of cobalt is the preparation of blue pigments. The substances met with in commerce for this purpose are cobalt ore—in various states, either raw, or, when roasted, under the name of *zaffre*—and smalt, which is a glass coloured with cobalt.

The ores in their native state contain sulphur, and the roasted as well as the raw ores, arsenic, iron, copper, lead, and bismuth. The following process may be adopted for the analysis and valuation of these substances.

A weighed quantity, reduced to very fine powder, mixed with two parts nitrate of potash and two parts anhydrous carbonate of soda, is gradually heated in a platinum or porcelain crucible until the whole is in quiet fusion. The cooled mass is then treated with warm water, which dissolves alkaline sulphate and arsenate, to some extent also, silicate. The solution is separated by filtration, and, if necessary, preserved for the estimation of sulphur and arsenic.

The insoluble portion is treated with hydrochloric acid, the solution evaporated to dryness at 212° F., dilute hydrochloric acid added, and the insoluble residue, consisting of silica and sand, collected on a filter.

The filtrate—containing the cobalt together with other metals—is treated with sulphuretted hydrogen until no further precipitate is formed, the sulphuret precipitate separated by filtration, and, if desirable, examined as directed in Chapter X.

The filtrate, then containing only cobalt, nickel, and iron, is carefully mixed with a solution of carbonate of soda till *nearly* neutralized, acetate of soda added, and the whole boiled. By this means the iron is separated. For the separation of the nickel and cobalt, they are both precipitated as carbonates by adding an excess of carbonate of soda; the precipitate collected on a filter, washed, and treated with a solution of oxalic acid. The oxalates of cobalt and nickel are then dissolved in solution of ammonia, and the liquid left for some days in an open porcelain basin, when, by the evaporation of ammonia, the nickel salt is gradually deposited. The cobalt solution is then poured off, evaporated to dryness in a small crucible, and the residue ignited and weighed.

The distinctive character of cobalt compounds is the fine blue colour which they communicate to borax when fused with it. Their colour is likewise characteristic in most instances.

Nickel and its compounds.

The native arsenide of nickel and the spiess obtained in the preparation of smalt are the chief sources from which nickel is procured. These may be analysed in the same manner as cobalt ores.

The compounds of nickel are not employed in the arts, but the metal, alloyed with others, is largely used as a substitute for silver.

The distinctive characters of nickel compounds in solution are, the production with caustic potash of a pale green precipitate (NiO,HO), insoluble in excess of the reagent, and the production of a greenish-white precipitate with ferrocyanide of potassium.

Nickel may be estimated volumetrically by means of the reaction between the peroxide and protochloride of tin,—

$$Ni_2O_3 + SnCl = Ni_2Cl_2 + SnCl_2,$$

the equivalent of tin in each experiment being estimated according to the method described at p. 134.

For this purpose the compound, dissolved in acid, is treated with potash and hypochlorite of lime, and the oxide formed (Ni_2O_3), collected on a filter, and reduced with the solution of protochloride of tin.

The quantity of substance to be taken for the experiment is 5·92 grms., or a fraction of that quantity, and the amount of nickel x is calculated by the proportion,

$$x : Sn = Ni_2 : Sn.$$

Compounds of arsenic.

Arsenious acid, AsO_3. 99.

Occurs in the form of a brittle colourless mass, or as a white powder. It is sparingly soluble in water, more soluble in dilute hydrochloric acid. The probable impurities are,—

Sulphates of baryta or *lime*, which are indicated, when the acid is sublimed in a test tube, by a fixed residue, which may be further tested in the usual manner.

Arsenate of potash, $KO(HO)_2,AsO_5$.

Should be readily and perfectly soluble in water; nitric or hydrochloric acid should not cause a precipitate.

Nitrate of potash may be detected by testing with solution of indigo and sulphuric acid.

Tersulphuret of arsenic, AsS_3. 123.

Known under the name of orpiment. The usual impurities are,—

Arsenious acid, detected by boiling with dilute hydrochloric acid, and testing the liquid with sulphuretted hydrogen.

Sand, clay, chalk, &c., remain undissolved by caustic soda.

Bisulphuret of arsenic, AsS_2. 147.

Should be perfectly volatilizable.

The distinctive characteristic of arsenic compounds is, that when heated with soda upon charcoal in the blowpipe flame, they evolve a peculiar alliaceous odour. Arsenic may likewise be tested for in solution by sulphuretted hydrogen, and the yellow precipitate examined as above for confirmation.

For the detection of minute quantities of arsenic, Marsh's test may be applied. It is based upon the conversion of arseniuretted hydrogen into arsenious acid when burnt, or,

under certain conditions, the separation of metallic arsenic from the burning gas.

Metallic zinc and dilute sulphuric acid are mixed in a small glass flask, furnished with a long bent tube, drawn out to a capillary opening at one end, and fitted into the flask by a cork. After the generation of hydrogen has continued for some minutes, the substance to be examined for arsenic is introduced, and the cork fitted tightly in. The gas issuing from the tube is then lighted, and a small porcelain capsule held close over the flame for a few seconds at a time. When arsenic is present, it is deposited upon the porcelain in the form of a black spot with a brilliant metallic lustre, which is dissolved in nitric acid, and the usual tests applied. Or a small beaker may be held over the flame at a greater distance, and then the arsenic is deposited as a sublimate of arsenious acid, which may be dissolved and tested as above.

In both cases the arsenic must be in solution in an oxidized state. When it is supposed to exist as sulphuret, the substance to be tested should first be melted with carbonate of soda or dissolved in nitric acid.

It is, of course, indispensable that both the zinc and sulphuric acid used should be pure, and, for the sake of certainty, the hydrogen generated should always be lighted and tried with the porcelain capsule, before adding the substance to be tested.

It is unfortunate that this test, though capable of indicating very minute quantities of arsenic, is to some extent inapplicable when antimony is present, since this substance behaves in a similar manner. When there is reason to suspect the presence of antimony, the metallic mirrors on the porcelain should be exposed over a glass containing sticks of phosphorus, partly immersed in water; the arsenical spots then disappear in a few hours, while those produced by antimony remain much longer unaltered. The analyst should familiarize himself with these reactions by comparative experiments.

Estimation of arsenic.

In the estimation of arsenic in alloys three cases may occur, for each of which a different method must be adopted.

1. When arsenic is to be separated from gold or platinum,

the substance is treated either with nitric acid, which dissolves the arsenic and other metals, or, when tin or antimony is also present, with nitro-hydrochloric acid, the platinum or gold being precipitated as directed at pp. 137, 138.

2. When arsenic is to be separated from silver, mercury, lead, bismuth, zinc, iron, or nickel, the substance in fine powder, or the dry saline residue left on evaporating the solution, from which gold or platinum have been separated, is ignited with twice its weight of carbonate of soda, and two and a half times its weight of nitrate of potash, by which means the arsenic is rendered soluble in water. The solution, when free from antimony or tin, is mixed with some nitric and hydrochloric acids, boiled, a solution of tartaric acid added in excess, then ammonia and sulphate of magnesia, which precipitate the arsenic acid as arsenate of ammonia and magnesia. The precipitate is collected upon a filter, washed with ammoniacal water, dried at 212° F., and weighed. It contains 41·4 per cent. of arsenic.

3. When arsenic is to be separated from tin or antimony, the substance is treated with nitric acid, excess of which is afterwards removed by evaporation. The dry oxides are then melted with six or eight times their weight of hydrate of soda in a silver crucible, the cooled mass treated with dilute alcohol, which dissolves tin and arsenic. The solution is evaporated to dryness, and treated with water, which dissolves only arsenic as arsenic acid, which is estimated as arsenate of ammonia and magnesia as above.

The results obtained in this manner do not indeed possess extreme accuracy, but they will in most instances be quite adequate to the objects desired, if the manipulation is carefully conducted.

Arsenious acid may be estimated volumetrically by means of a solution of hypochlorite of lime, according to the method described for the valuation of that substance (p. 97).

Compounds of tin.

Oxide of tin, SnO_2. 74·8.

This substance is known in commerce under the name of putty powder. It generally contains the impurities found in metallic tin, and is sometimes adulterated with

Oxide of lead, detected by its solubility in dilute nitric acid.

Chalk, indicated by effervescence with acids.

Sulphates of baryta or *lime* may be detected by their insolubility in caustic soda, which dissolves oxide of tin.

Chloride of tin, SnCl + 2HO. 112·3.

Occurs in commerce in a solid form under the name of tin salt. It should be perfectly soluble in water mixed with a little hydrochloric acid or chloride of ammonium. The impurities which are of most consequence are,—

Copper, lead, zinc, and *iron*, which may be detected by treating the solution with sulphuret of ammonium containing sulphur, and digesting the precipitate in an excess of the reagent, which dissolves the sulphuret of tin and leaves the other sulphurets in the form of a black residue.

Alkaline chlorides, with which the salt is sometimes adulterated, may be detected by precipitating the tin by sulphuretted hydrogen and evaporating the filtrate to dryness. In the absence of such substances it should not leave any residue.

Perchloride of tin, $SnCl_2$. 129·8.

Generally in the liquid state, and may be tested in the same manner as the chloride.

Stannate of soda, $NaO,SnO_2 + 3$ aq. 132·8.

This salt should be perfectly soluble in water, and on the addition of dilute nitric acid to the solution, a precipitate should be formed at once, without effervescence, which would indicate the presence of *carbonate of soda*.

The distinctive character of tin and most of its compounds is, that when treated with nitric acid, the white insoluble oxide, SnO_2, is formed, which, when heated with soda upon charcoal in the blowpipe flame, gives metallic beads without any incrustation (Chap. X.). Tin compounds in solution give with sulphuretted hydrogen, in the presence of free acid, a brown or yellow precipitate; the former reaction is indicative of protosalts, the latter of the persalts of tin.

Estimation of tin.

The estimation of tin is frequently effected by means of the insolubility of its oxide (SnO_2) in excess of nitric acid. This compound contains 78·61 per cent. of tin. When the tin is in solution in a state of combination corresponding to

SnO_2, it is precipitated by boiling with an excess of carbonate of ammonia. Protosalts of tin are to be converted into persalts by previous treatment with chlorine. Metallic substances may be heated with moderately strong nitric acid until the oxidation is complete, and the binoxide collected by filtration, washed and weighed. The details of the process are given at p. 136.

Volumetrical estimation of tin.

The process by which this is effected resembles the estimation of iron, inasmuch as tin forms with chlorine two compounds, SnCl and $SnCl_2$, which are mutually convertible.

The reagent used for the purpose is bichromate of potash, and the reaction is represented by the equation—

$$3SnCl + 2CrO_3 + 6HCl = 3SnCl_2 + Cr_2Cl_3 + 6HO.$$

The point at which the reaction is completed is indicated by the intense blue colour produced by starch and free iodine, and as the liberation of iodine from iodide of potassium by bichromate of potash does not take place in the presence of protochloride of tin, a few drops of iodide solution and some starch-paste are mixed with the tin solution before adding the reagent.

The normal solution is made by dissolving 41·6 grms. of bichromate in water to the volume of one litre; each cubic centimeter will be equivalent to 0·1 grm. of tin, and this quantity multiplied by the number of cubic centimeters (C_n) required to complete the reaction, gives the amount of tin.

Quantity of material. Per-centage results may be obtained directly by operating with 5·0 grms. of the substance in which tin is to be estimated.

Experiment. For the valuation of protochloride of tin in solution, a known volume may be taken; of the crystallized salt an average sample is selected, and the weighed quantity dissolved in water with addition of hydrochloric acid; a little solution of starch and a few drops of iodide of potassium solution are mixed with it, and the normal solution added from the volumeter until the blue colour appears.

In the case of alloys and other substances soluble in hydrochloric acid, the solution of the weighed quantity is boiled with iron wire, by which means perchloride of tin is

reduced to the state of protochloride,, while arsenic, antimony, copper, lead, and mercury are precipitated at the same time in the metallic state.

Alloys, &c., insoluble in hydrochloric acid, are dissolved in nitro-hydrochloric acid, and the perchloride of tin reduced by boiling with iron wire. Even the oxide of tin, formed by treatment with nitric acid, is dissolved as protochloride by continued digestion with strong hydrochloric acid and iron wire.

The protochloride of iron, formed by this treatment, does not affect the reaction of the bichromate with the tin, because it is not converted into perchloride by that reagent in the presence of protochloride of tin.

The reaction of protochloride of tin with bichromate of potash, and the powerful as well as definite reducing action of protochloride of tin, renders this process applicable to the indirect estimation of a number of other substances, in a manner similar to that by means of the chloride of iron and the normal solution of permanganate.

The general principle of these indirect estimations is essentially the same as that involved in the permanganate process; the material in which some substance is to be estimated is treated with a known quantity Q of protochloride of tin in excess, and the quantity of tin, T, converted into perchloride, ascertained by estimating, with the normal solution of bichromate, the quantity of tin t still remaining in the state of protochloride; then,—

$$T = Q - t.$$

The amount of the substance sought is then calculated by means of the equation representing the reaction for the particular case.

The details of the experiments, and the formulæ for calculating the results, are given under the head of the substances that may be estimated in this manner.

The substances to which this process is applicable are enumerated below, together with the equations by which their amount is found.

Chromic acid	x	: T =	$2CrO_3$	:	Sn_3.
Oxide of manganese	x	: T =	MnO_2	:	Sn.
Ferricyanide of potassium	x	: T =	(K_3Cfy_2)	:	Sn.
Nickel.....	x	: T =	Ni_2	:	Sn.

Tin	x : c	=	Sn_3	: Cr_2.
Copper	x : c	=	Cu_6	: Cr_2.
Lead	x : T	=	Pb	: Sn.
Mercury	x : T	=	Hg	: Sn.
	x : T	=	Hg_2	: Sn.
Chlorine	x : T	=	Cl	: Sn.
Iodine	x : T	=	I	: Sn.

Antimony and its compounds.

Sulphuret of antimony, SbS_3. 177.

Known in commerce under the name of crude antimony. It is a dark gray mass with a radiated crystalline structure, yielding a blackish-brown powder, and easily fusible (thus distinguished from manganese). It should be perfectly soluble in hydrochloric acid, and the solution should not become turbid on cooling (indicative of *lead*). When melted with four times its weight of nitrate of potash, it should yield a white mass; *iron* or *lead* are indicated by a yellow colour of this mass. Arsenic is frequently present, and is to be tested for by fusion with soda upon charcoal by means of the blowpipe, when the characteristic garlic odour is evolved. See p. 129.

Chloride of antimony, $SbCl_3$. 235·5.

The pure salt is white and semi-solid, but it is generally met with in commerce as an acid, oily solution, sometimes of a dark brown colour, under the name of butter of antimony. Its density varies from 1·2 to 1·5. With water it gives a copious white precipitate.

The distinctive character of antimony compounds in solution is the formation, with sulphuretted hydrogen in the presence of free acid, of an orange precipitate, SbS_3, soluble in sulphuret of ammonium. When oxidized by means of strong nitric acid, antimony, like tin, gives a white insoluble residue, which may, however, be distinguished from the binoxide of tin by its giving brittle globules and a white incrustation when fused with carbonate of soda on charcoal in the reducing blowpipe flame.

Estimation of antimony.

In the case of soluble compounds corresponding to the teroxide (SbO_3), this metal may be estimated as sulphide, by

treating the acid solution with sulphuretted hydrogen and weighing the precipitate (SbS_3), dried at 212° F.

In the case of compounds corresponding to the higher oxide (Sb_2O_5), it may be estimated as antimonious acid by evaporating the solution to dryness with a large excess of nitric acid, and weighing the ignited residue (SbO_4).

The method of estimating antimony in alloys varies according to their composition:—

1. When the alloy is free from tin, a weighed quantity is heated with nitric acid until the whole of the metal is oxidized, and the excess of acid evaporated off. The dry residue is ignited, digested with very dilute nitric acid, collected on a filter, washed, and weighed as antimonious acid, containing 80·1 per cent. of the metal.

2. When the alloy contains tin, but no other metals, it is dissolved in hydrochloric acid by means of the gradual addition of nitric acid, the diluted solution digested at a moderate heat with a strip of clean pure tin, and hydrochloric acid added from time to time. The antimony is thus precipitated, after a time perfectly, as a black powder, which is collected upon a weighed filter, washed, dried, and weighed.

3. When the alloy contains, besides antimony and tin, other metals, as lead, bismuth, &c., it must be treated with nitric acid as in 1, and the mixture of antimony and tin oxides weighed. In the solution of another quantity of the alloy, the tin is estimated by the volumetrical method (p. 133), and from these data the amount of antimony may be calculated:—

If, for instance, a given weight of the alloy gives 1·32 grm. of a mixture of binoxide of tin with antimonious acid, and the volumetrical experiment with an equal weight of the alloy indicates 0·84 grm. of tin, the alloy would contain 0·20 grm. of antimony; for the quantity of binoxide of tin y equivalent to 0·84 grm. of tin is

$$58\ (Sn) : 74\ (SnO_2) = 0{\cdot}84 : y\ (= 1{\cdot}07\ \text{grm.}),$$

and this, deducted from the weight of the mixed oxides, gives $1{\cdot}32 - 1{\cdot}07 = 0{\cdot}25$ grm. as the weight of the antimonious acid, equivalent to 0·20 grm. of the metal.

Compounds of platinum.

Bichloride of platinum, $PtCl_2$. 169·7.

Is generally met with in solution, which, when evaporated, leaves a reddish-brown mass, soluble in alcohol, æther, and water. It forms with potash and ammonia salts, yellow compounds, very sparingly soluble in water and insoluble in alcohol. When the solution of bichloride of platinum in alcohol is mixed with a slight excess of chloride of ammonium, the filtered liquid should not leave any residue when evaporated to dryness, and ignited to drive off the ammoniacal salt.

The distinctive character of platinum compounds in solution is the formation with chloride of potassium or ammonium of the yellow, sparingly soluble compounds. Platinum is always estimated by weighing the metal left on igniting the double chloride of platinum and ammonium.

Gold and its compounds.

Chloride of gold, $AuCl_3$. 303·5.

The crystallized salt has a pale yellow colour, is deliquescent, and readily soluble in water, alcohol, and æther. The solution stains the skin deep purple. The amount of gold is estimated according to the method described below, and the presence of alkaline chlorides may be detected by strongly heating a portion of the salt so as to decompose it, and then treating the residue with water, which would dissolve the alkaline chlorides.

Purple of Cassius, $Au_2 + 3SnO_2 + 4HO$.

This substance is used for painting glass and porcelain. The proportion of gold and the presence of any foreign substance may be ascertained by igniting a weighed quantity, treating it with nitro-hydrochloric acid, separating the oxide of tin by filtration, and estimating the gold in the filtrate.

The amount of gold in this substance varies greatly, from 24 to 79·5 per cent. The loss by ignition should not amount to more than about 8 per cent.

The solutions used for galvanoplastic purposes consist of,—chloride of gold with *a*, bicarbonate of potash; *b*, phosphate of soda; *c*, hyposulphite of soda; *d*, cyanide of potassium, or *e*, ferrocyanide of potassium. The amount of gold in

these solutions may be ascertained by evaporating to dryness a known quantity, igniting the residue with twice its weight of chloride of ammonium until no more vapour is given off. The mass is treated with dilute hydrochloric acid, the insoluble portion consisting of gold—perhaps with oxide of iron—dissolved in nitro-hydrochloric acid, and the gold estimated by precipitation.

The solvents *a*, *c*, and *d* are recognized by effervescence with acids; the two latter, and also *e*, are distinguished by the odour of sulphurous or hydrocyanic acid evolved. *b* may be recognized by the precipitate formed on the addition of ammonia and sulphate of magnesia to the solution obtained after ignition with chloride of ammonium.

The distinctive characters of gold compounds in solution are,—1, the precipitation by sulphate of iron of the metal in the state of a bluish powder, which acquires metallic lustre when rubbed (the liquid to which this test is applied must not contain free nitric acid); 2, the formation of a reddish-brown precipitate, purple of Cassius, on the addition of a solution of protochloride of tin containing a little perchloride.

Estimation of gold.

This metal is always estimated in the metallic state. It may be precipitated from solution, for this purpose, by means of protosulphate of iron, digested with weak hydrochloric acid, collected on a filter and weighed.

The value of alloys containing gold in any considerable proportion is, however, always estimated by a special process, and with arrangements that admit of a great number of results being obtained within a very short time.

The separation of copper from gold for this purpose is always effected by melting the alloy with lead upon a small cup of compressed bone-ash, and allowing a gentle current of air to pass over the surface of the melted mixture. A rapid oxidation of the copper and lead takes place, and gold being unoxidizable under these conditions, it remains upon the cupel in the form of a bright globule.

The action of lead in this process is twofold:—it facilitates the fusion of the alloy by acting the part of a solvent to it at a temperature much below the melting-point of either gold or copper, and the oxide of lead formed simultaneously

with the oxide of copper acts likewise as a solvent to this otherwise very infusible substance. This solution of oxide of copper in melted oxide of lead is rapidly absorbed by the porous cup of bone-ash, and when the temperature and general conditions of the experiment are properly managed, may thus be separated from the gold.

The proportion of lead requisite for the separation of copper from gold depends upon the amount of copper in the alloy, for when the oxide of lead contains more than a certain proportion of oxide of copper, it is too viscid for absorption into the cupel. The proportions of lead for alloys of different value are given in the table:—

Decimal value of the alloy in gold.	Proportion of lead for cupellation.
1000	—
950	10
900	14
800	20
700	24
500 and less	34

The separation of silver from gold is always effected by treatment with nitric acid; but for this purpose the silver must bear a certain proportion to the gold, not less than twice as much, otherwise the whole of the silver would not be dissolved, nor more than three times as much, because then the gold would be reduced to the state of powder, and its collection rendered more tedious, so that in the assay of an alloy containing a smaller proportion of silver, it is necessary to add enough silver, judging from the probable value of the alloy, to make up this proportion. This is done by cupellation, because the fusion of the metals is facilitated by lead, and the copper that is generally present in gold alloys is removed at the same time; and when the button of gold and silver thus obtained is flattened, rolled into a loose coil and boiled in nitric acid (of 1·300), the silver is dissolved without the gold being disintegrated, a circumstance very favourable for its subsequent weighing.

It rarely happens that alloys of gold with copper do not contain some amount of silver, and as the behaviour of silver on cupellation with lead is the same as that of gold, the

button obtained by simple cupellation with lead would in such case contain the silver. In order therefore to estimate the amount of gold, it will be necessary to have recourse to the parting process, which, indeed, it is advisable always to adopt; and when it is requisite, a separate experiment is made for the purpose of ascertaining the amount of silver in the alloy.

Bismuth and its compounds.

Trisnitrate of bismuth, BiO_3, NO_5, HO.

Known under the name of pearl-white; its composition varies, but it should be pure white, and when heated evolve red nitrous vapour, at the same time becoming yellow, but not black. It should be perfectly soluble in nitric or hydrochloric acid.

The probable impurities are,—

Carbonate of bismuth, indicated by effervescence.

Iron, sulphuric acid, and *lead*, indicated respectively in the hydrochloric solution by K_2Cfy, BaCl, and SO_3.

Oxide of bismuth, BiO_3. 232.

A pale yellow powder, which becomes darker when heated, and should be perfectly soluble in acids. It is tested as above.

The distinctive character of bismuth and its compounds is, that they yield, when fused with carbonate of soda on charcoal in the reducing blowpipe flame, brittle globules and a yellow incrustation.

Estimation of bismuth.

As there is no characteristic reaction by which this metal may be separated from others, the methods adopted for its estimation in alloys, &c. must be adapted to the particular cases indicated by the previous qualitative examination.

The metals usually associated with bismuth in alloys are copper, tin, antimony, lead, mercury. By treatment with nitric acid, antimony and tin are separated in an insoluble form. Lead may be separated from the solution by sulphuric acid, then evaporating to dryness, redissolving in very dilute sulphuric acid, and collecting the sulphate of lead by filtration. A solution containing copper and bismuth may be

mixed with excess of carbonate of ammonia, which after some time dissolves the copper, but not the bismuth.

For the separation of bismuth from mercury, the nitric solution is boiled with excess of carbonate of ammonia, the precipitate of bismuth collected by filtration, and weighed as oxide after ignition.

Copper and its compounds.

Sulphate of copper, $CuO,SO_3,+5HO$. 124·7.

Pure blue crystals without any green tint, perfectly soluble in water. The most frequent impurities are,—

Sulphates of iron and *zinc*. The presence of iron is detected by warming the solution with a few drops of nitric acid, and then adding carbonate of ammonia in sufficient excess to redissolve the precipitate. When oxide of iron is present, it remains in the form of a brown precipitate, and may be collected and estimated. Zinc would remain dissolved in the ammoniacal liquid, and may be detected by boiling it with caustic soda until the smell and colour disappear, separating the oxide of copper by filtration, and treating the filtrate with sulphuretted hydrogen; when zinc is present, it is precipitated as sulphuret, and may be estimated as directed at p. 109.

Pure sulphate of copper should contain 31·8 oxide of copper (CuO), or 25·3 copper.

Nitrate of copper, $CuO,NO_5,+3HO$. 120·7. } are both tested in the same manner as the sulphate.

Sulphate of copper and ammonia, $CuO,SO_3\,NH_4O,SO_3$, }

Carbonates of copper, { *a*. $2CuO,CO_2+CuO,HO$, *m*. CuO,CO_2+CuO,HO.

The native carbonates, azurite (*a*) and malachite (*m*), as well as the corresponding artificial carbonates, *mountain blue*, *Bremen blue*, or *blue verditer*, and *Bremen green*, are mixtures of carbonate of copper, with oxide of copper and water in variable proportions, and are met with in commerce as pigments, sometimes containing more or less earthy matter—chalk, carbonate of magnesia, alumina, &c.—glue, starch, &c. The latter may be detected by digesting the substance in hot water, and evaporating the liquid. The portion inso-

luble in water should be perfectly dissolved by hydrochloric acid, and on the addition of carbonate of ammonia in sufficient excess to redissolve the carbonate of copper, the earthy admixtures will remain undissolved; they may be collected by filtration and estimated. Oxide of zinc is to be tested for in the ammoniacal solution as in the case of sulphate of copper.

Oxide of copper, CuO. 39·7.

The pure oxide should not give off any red nitrous vapour or smell when heated; it should be perfectly soluble in dilute nitric acid, and the precipitate formed on adding carbonate of ammonia to the solution should dissolve completely in an excess of the reagent. The probable impurities are,—

Oxide of iron, indicated by brown flocks, which remain undissolved when an excess of carbonate of ammonia is added to the solution.

Earthy substances would likewise remain undissolved on applying the above test. The precipitate may be further examined as directed in Chapter X.

Hydrated oxide of copper.

Occurs in commerce as a pigment under the name of Bremen blue, and is tested in the same manner as the previous substances. It frequently contains an admixture of glue, which may be separated by warm water, and estimated by evaporating the solution to dryness.

Arsenite of copper, CuO,AsO_3 (*a*), and

Arsenio-acetate of copper, $CuO,\bar{A}+3CuO,AsO_3$ (*b*).

Known in commerce under the names of Scheele's green and Schweinfurt green, and used as pigments. They are insoluble in water, but soluble both in acid and ammonia solution. Schweinfurt green evolves a very peculiar and offensive odour when heated in a test-tube; Scheele's green evolves only the characteristic odour of arsenic. (The vapours in both cases are very poisonous.) The proportions between the arsenious acid and oxide of copper vary much.

The probable impurities are,—

Carbonate of copper, indicated by effervescence with acids.

Clay, heavy spar, gypsum, remain undissolved by acids, and

Earthy substances, are detected as in the case of the carbonates of copper.

Chromate of lead, with which they are sometimes adulterated, may be detected by dissolving in hydrochloric acid, adding water and sulphuric acid, which precipitates the lead. The filtrate from the sulphate of lead is mixed with alcohol, boiled, and ammonia then precipitates green oxide of chromium.

Acetates of copper, { *a*. $CuO\bar{A}HO$. *b*. $CuO\bar{A} + CuOHO + 5HO$.

The neutral crystallized salt *a*, erroneously called *distilled* verdigris, should be perfectly soluble in water; the basic salt *b*, ordinary verdigris, is only partially soluble in water, but should dissolve perfectly in acetic acid. The impurities are most likely to be mechanical.

Fragments of copper and vegetable substances remain undissolved.

The solutions used for galvano-plastic purposes consist of,—

a. Copper salts, dissolved by cyanide of potassium.

b. Carbonate of copper, dissolved by tartrate of potash and carbonate of potash.

c. Hydrated oxide of copper, dissolved by sulphite of soda.

The nature of the solvent may be ascertained by the addition of sulphuric acid, with which *a* disengages the odour of hydrocyanic acid, *c* sulphurous acid; tartrate of potash is detected by adding hydrochloric acid and evaporating the liquid, when bitartrate of potash separates.

The solution *b* has always, and *a* generally, an alkaline reaction. The amount of copper in any one may be ascertained by evaporating a certain quantity to dryness, and igniting the residue with twice its weight of chloride of ammonium. The mass is then dissolved in nitric acid, warmed, and the solution filtered. An insoluble residue may consist of oxide of iron from ferrocyanide. The copper is estimated as directed below.

The distinctive character of copper compounds is the intense blue colour produced when ammonia is added in excess to the solution.

Estimation of copper.

The most advantageous method is probably that based upon the definite reaction between suboxide of copper and perchloride of iron in the presence of hydrochloric acid,

$$Cu_2O + Fe_2Cl_3 + HCl = 2CuCl + 2FeCl + HO.$$

The experiment is carried out in the following manner. The weighed copper compound or alloy is dissolved in water or hydrochloric acid, the solution introduced into a capacious porcelain or platinum dish, and mixed when cold with a solution of tartrate of potash and excess of caustic potash. The deep blue liquid thus obtained is mixed with an aqueous solution of grape-sugar (honey), or still better milk sugar, in sufficient quantity, and the whole heated upon a water-bath to 176° or 194° F. until the liquid begins to assume a brownish colour at the edges,—a sign that all the copper is precipitated, and that the reaction between the sugar and potash has commenced.

When the precipitate has settled, which takes place very readily, it is collected upon a paper filter; the liquid filtered through is almost opake, and at the surface of contact with the wash water there sometimes appears to be a yellowish turbidity, which, however, is not owing to the presence of suboxide of copper, and disappears on mixing the liquids. The precipitate must be washed with hot water until the filtrate is colourless. Sometimes, and especially when the mixture has been much heated, the suboxide has a dirty colour, but this is merely owing to its being in a rather denser state, and has no influence whatever upon the experimental result.

It frequently happens that some of the suboxide of copper adheres to the sides of the flask or basin, but this is of no consequence, as the same vessel is used for the treatment with perchloride of iron.

By gently heating the mixture of suboxide and perchloride, the subchloride of copper at first formed readily dissolves. The green liquid is then filtered into a capacious flask, the filter thoroughly washed, a little hydrochloric acid added, and then water until the temperature is about 96° F. The quantity of protochloride formed is then estimated by means of a solution of permanganate.

When other metals are present in the substance analysed, they may either be separated before the precipitation of the suboxide, or remain in solution after that has been effected, when they are removed by mere filtration. Silver and mercury may be separated as chloride, or equally well with chloride of mercury by potash, even in the presence of tartaric acid. The other oxides soluble in potash are not precipitated, with the exception of oxide of nickel, and that only partially, and as it has no reducing action, the result is not affected. Peroxide of iron dissolves in potash and tartaric acid, giving a brown solution, which deposits peroxide after long boiling, but not when heated only to 212° F.; and even if the suboxide of copper should be mixed with a small quantity of peroxide of iron, it would merely give rise to the formation of a little more perchloride, without in any way influencing the result.

Colorimetric method.

All the salts of oxide of copper, when mixed with an excess of ammonia, acquire a blue colour, the intensity of which is proportionate to the quantity of oxide dissolved.

This fact has been applied to the estimation of copper in the following manner. A normal solution is prepared by dissolving 0·500 grm. of pure copper in nitric acid, adding an excess of ammonia and water to the volume of one litre. Of this solution 5 cubic centimeters are introduced into a glass tube, which is then sealed by the blowpipe.

2·000 grms. of the alloy to be examined are then dissolved in nitric acid, mixed with an excess of ammonia and water to the volume of 100 cubic centimeters.

5 cubic centimeters of this solution are then introduced into a tube of the same width as that containing the normal solution, and graduated into cubic centimeters. A sheet of white paper is then placed behind the two tubes, and water added to the liquid until it acquires the same shade of colour as the normal solution. In order to ascertain this point more exactly, the observations are made through a blue glass.

When, for example, the 5 cubic centimeters require to be diluted to 30 cubic centimeters before the colour is of the same intensity as the normal solution, then every 5 cubic

centimeters will contain as much as 5 cubic centimeters of the normal solution, that is 0·0025, consequently the 30 cubic centimeters, or the 5 cubic centimeters of the alloy solution, contain 0·0025 × 6 = 0·0150 grm. of copper; the 200 cubic centimeters of alloy solution, or the 2·000 grms. of alloy, 0·600 copper. The alloy will therefore contain 30 per cent. copper.

The presence of nickel or cobalt in the alloy renders the method inapplicable, and the colour of the normal solution appears to alter somewhat when it is kept.

Lead and its compounds.

Oxide of lead, PbO. 111·7.

Occurs in commerce under the name of litharge; it should be perfectly soluble in nitric acid without effervescence.

The probable impurities are,—

Carbonate of lime, may be detected by dissolving the litharge in dilute nitric acid, precipitating the lead by sulphuretted hydrogen, and treating the filtrate with oxalate of ammonia.

Copper, detected by digesting the substance in ammonia.

Earthy substances, either remain undissolved by dilute nitric acid, or may be detected in the liquid from which the lead has been separated by sulphuretted hydrogen.

Red oxide of lead, $2PbO+PbO_2$. 343·1.

Only partially soluble in dilute nitric acid; in other respects resembles litharge.

Carbonate of lead, PbO,CO_2+PbO,HO. 133·7.

The substance met with in commerce as a pigment varies in composition; it sometimes contains basic acetate of lead, and is known under several names. The amount of carbonate may be ascertained by means of the apparatus (fig. 33). The amount of acetic acid may be ascertained by distilling a weighed quantity with dilute sulphuric acid, and estimating the acetic acid in the distillate by means of normal solution of alkali, &c. The probable impurities are,—

Sulphates of baryta, lead or *lime*, all of which remain undissolved by dilute nitric acid, and may be further examined as directed in Chap. X.

Carbonates of lime or *zinc* are detected in the hydrochloric solution after precipitating lead by sulphuretted hydrogen.

Zinc is precipitated by sulphuret of ammonium, and in the filtrate, lime is precipitated by oxalate of ammonia.

Phosphate of lime—from adulteration with bone-ash—is detected by treating the nitric solution with an excess of caustic soda, which, unless phosphate or carbonate of lime is present, should perfectly redissolve the precipitate first formed; or, by evaporating the nitric solution to dryness on a water-bath, and digesting the residue with alcohol, which dissolves acid phosphate of lime.

Acetate of lead, PbO,A + 3HO. 189·7.

Known in commerce under the name of sugar of lead. The pure salt should be perfectly white, soluble in water, and the liquid remaining after precipitating the lead by sulphuretted hydrogen should not leave any residue on evaporation.

The distinctive characters of lead compounds in solution, are the production with sulphuric acid of a white precipitate (PbO,SO_3), and with sulphuretted hydrogen of a blackish-brown precipitate (PbS) in acid solutions.

Estimation of lead.

The insolubility of sulphate of lead admits of the estimation of this metal gravimetrically with tolerable accuracy. The solution containing the lead is mixed with a slight excess of dilute sulphuric acid, then with alcohol, the precipitate (PbO,SO_3) collected on a filter, washed with alcohol, dried, and weighed.

Among the volumetrical methods, which are all somewhat operose, that based upon the reaction between binoxide of lead and protochloride of tin is probably the best:—

$$PbO_2 + SnCl + 2HCl = PbCl + SnCl_2 + 2HO.$$

The amount of lead x in a given quantity of material is found by ascertaining, as described at p. 133, how much (T) of a known quantity of tin, in the state of protochloride, is converted into perchloride by that lead, previously converted into binoxide:—

$$x : T\ (= Q - t) = Pb : Sn.$$

The lead to be estimated is converted into binoxide by means of hypochlorite of lime.

Experiment. A solution of the material in water or acid is mixed with an excess of potash, solution of hypochlorite

of lime added in excess, and the whole heated to 208° F. for some time, until the whole of the lead is peroxidized. Galena is in the first instance oxidized by nitric acid, and the sulphate of lead, dissolved in potash, treated in the same manner.

The precipitate of binoxide is collected upon a filter, washed with boiling water, and transferred into a wide-mouthed flask by breaking the filter with a glass rod, and washing off adherent portions, first by the protochloride of tin and then by hot water. Hydrochloric acid is then added in excess, and heat applied until perfect solution is effected.

The clear liquid is poured into a beaker, mixed with iodide of potassium and starch, and the excess of protochloride of tin estimated with the normal solution of bichromate of potash.

The reaction between chromate of lead and protochloride of iron,—

$$2PbO,CrO_3+6FeCl+8HCl=2PbCl+Cr_2Cl_3+3Fe_2Cl_3+8HO,$$

is likewise available for the estimation of lead. The entire quantity of lead in a solution may be readily converted into neutral chromate (PbO,CrO_3), which, on the application of a gentle heat, settles rapidly, and may be collected and washed with little trouble. Even sulphate of lead, at least when freshly precipitated, is completely converted into chromate when digested with an excess of bichromate of potash.

The amount of lead x is found by ascertaining, as described at p. 124, how much of a known quantity of iron, f, in the state of protochloride is converted into perchloride by the chromate,

$$x : F\ (= f - f) = Pb_2 : Fe_6.$$

Experiment. In the case of acid solutions an excess of bichromate is essential, because then the acid combines with the potash, and the solution or decomposition of the chromate of lead is prevented. The precipitate of chromate of lead is washed by decantation, mixed with a known quantity of protochloride of iron and an excess of hydrochloric acid.. At a moderate temperature, the chromate of lead dissolves rapidly and completely; the liquid assumes a green colour, and some chloride of lead separates in crystals. The liquid is rapidly filtered into a flask, the filter washed with hot water, and the residual protochloride of iron is estimated by means of the normal solution of permanganate, p. 123. The end of

the reaction is distinctly perceptible, notwithstanding the green colour of the solution.

The quantity of material to be taken for experiment in order to ascertain directly the per-centage of lead is, for metallic substances, 1·03 grm.

Among the metals usually associated with lead in alloys, tin and antimony are separated as insoluble oxides by the treatment with nitric acid; silver may be separated by hydrochloric acid sufficiently dilute not to cause precipitation of lead; mercury in compounds, corresponding to Hg_2O, may likewise be separated by hydrochloric acid, or better, by boiling the solution with nitric acid, so as to convert them into compounds corresponding to HgO, the chromate of which is soluble in acids.

Bismuth is the only metal which there is any difficulty in separating. The chromate of bismuth is soluble in concentrated nitric acid, but in the presence of the excess of bichromate of potash, requisite for the perfect precipitation of lead, basic chromate of bismuth is precipitated. The best, although not quite exact, method of separation is to boil the nitric solution of the metals with acetate of soda in excess, when the bismuth is for the most part precipitated, while lead remains in solution.

Lead may also be estimated by the reaction between permanganate of potash and oxide of lead dissolved in caustic potash:—

$$3PbO + MnO_7 = 3PbO_2 + 2MnO_2.$$

However, the reaction takes place only at 212° F., and towards the end of the experiment, when the quantity of lead is small, it is very sluggish, and on account of the irregularity and violence with which the evolution of vapour takes place, there is danger that the flask may be broken or the liquid thrown out.

Another method of estimating lead is based upon the reaction between this metal in solution and sulphuret of sodium:—

$$PbO + NaS = PbS + NaO.$$

Its principal advantage, without which indeed it would be valueless, consists in the circumstance that very few of the oxides of other metals, precipitable by sulphuret of sodium, are soluble in caustic potash. Zinc and arsenic

are the only metals whose oxides are soluble in potash that are likely to be associated with lead; the latter is not precipitated from an alkaline solution by sulphuret of sodium, and the former is not precipitated by sulphuret of sodium until the whole of the lead has been removed from solution.

The metals most frequently associated with lead—tin and antimony—may be separated by treatment with nitric acid. Bismuth alone would affect the accuracy of the results, but it is not likely to occur, except in special instances, and then it would be better to adopt the method first described.

Mercury and its compounds.

Chloride of mercury, Hg Cl. 135·5.

Should be colourless, translucent, perfectly soluble in two parts of boiling water and in alcohol. It should volatilize without leaving any residue.

Nitrate of mercury, $2HgO,NO_5+2$ aq. 288.

Decomposed by water, but perfectly soluble in dilute nitric acid. The solution should not give any precipitate with chloride of sodium (indicative of the lower nitrate). Ammonia should give a white precipitate.

Sulphuret of mercury, HgS. 116.

Known in commerce under the name of cinnabar or vermilion. The latter is most likely to be adulterated or to contain impurities. Pure sulphuret of mercury is not acted upon by dilute nitric acid, but when

Metallic mercury is present, it is dissolved, and the acid liquid gives a precipitate with sulphuretted hydrogen.

Nitrate of mercury is indicated by a darkening of colour on pouring a cold solution of sulphuret of sodium upon the substance.

Realgar is detected by boiling the substance with caustic soda, which dissolves the sulphuret of arsenic; on adding to the liquid a slight excess of nitric acid and then sulphuretted hydrogen, the arsenic is precipitated as sulphuret.

Dragon's-blood is detected by its solubility in hot alcohol.

Red lead, oxide of iron, &c. are indicated by a fixed residue after the application of a strong heat.

Iodide of mercury, HgI. 227·1.

A red powder, perfectly insoluble in water, soluble in

boiling alcohol, and is thus distinguished from vermilion. It should be perfectly volatilized by heat.

Subchloride of mercury, Hg_2Cl. 235·5. (*Calomel.*)

The pure substance should be volatilizable without residue, and when shaken with boiling water, the filtrate should not give any precipitate with sulphuretted hydrogen or potash, which would indicate the presence of chloride of mercury.

Subnitrate of mercury, Hg_2O,NO_5+2 aq. 280.

This salt is decomposed by water into an insoluble basic salt, and an acid salt which dissolves. It is soluble in weak nitric acid. Chloride of sodium should precipitate the whole of the mercury, so that caustic soda will not produce any precipitate in the filtrate. Caustic ammonia should produce a black precipitate, and not one that finally becomes white. These two reactions would indicate the presence of nitrate of mercury.

The distinctive character of mercury compounds is, that when heated in a long test-tube with a little moist carbonate of soda, a sublimate of metallic mercury is deposited on the cool portion of the tube.

Estimation of mercury.

The method to be adopted for the estimation of mercury in amalgams depends upon the nature of the metals with which it is associated. When these metals are neither volatilized nor oxidized by heat, the mercury may be estimated by the loss on ignition for some time in a porcelain crucible. When the accompanying metals are oxidized by heating in the air, the mercury must be distilled off in a small retort, made of glass tube, the neck of which is sealed as soon as the whole of the mercury has been driven off.

When the mercury is accompanied by a volatile metal, such as zinc, the above methods cannot be adopted. The material must then be dissolved in hydrochloric acid, a few drops of nitric acid being added from time to time, and the solution, afterwards boiled with an excess of hydrochloric acid so as to ensure the absence of nitric acid, mixed with protochloride of tin, and boiled for two or three minutes. By this means the mercury is precipitated in the metallic state; it may be collected by decantation, dried with filter paper, and weighed.

Silver and its compounds.

Nitrate of silver, AgO,NO_5. 170·1.

The pure salt forms transparent colourless crystals. It is also met with in sticks, which should be without any green colour, and perfectly soluble in an equal weight of water.

The probable impurities are,—

Copper, indicated on the addition of an excess of ammonia (see p. 143).

Nitrate of potash, with which the melted salt is sometimes adulterated, may be detected by precipitating the silver by hydrochloric acid, and evaporating the filtrate to dryness. When the salt is pure there should be no residue.

The silver solutions used for galvano-plastic purposes contain:—

a. Cyanide of silver, dissolved in cyanide or ferrocyanide of potassium.

b. Silver salts dissolved in mixtures of chloride of sodium and tartrate of potash; or, in hyposulphite of soda, and frequently mixed with alcohol and gallic acid; and solid mixtures containing chloride of silver are likewise used for plating.

The amount of silver in any of them may be ascertained by heating the dry substance (in the case of liquids, the residue obtained by evaporating a certain quantity) with twice its weight of powdered chloride of ammonium, until vapour is no longer given off, treating the residue with successive portions of ammonia solution until, on the addition of hydrochloric acid in slight excess, the liquid no longer becomes turbid, and then boiling the ammoniacal solution—containing the silver as chloride—with sugar and caustic potash or soda. The silver is by this means precipitated in a metallic state, and after thorough washing with boiling water, may be collected and weighed.

The distinctive character of silver compounds in solution, is the formation with soluble chlorides of a white curdy precipitate (AgCl), which becomes dark blue or black on exposure to light, and when freshly precipitated is soluble in ammonia.

Estimation of silver.

This metal may be estimated with great accuracy in the form of chloride. The solution is slightly acidulated with

nitric acid, hydrochloric added in slight excess, the whole warmed for a few minutes, and the chloride washed by decantation, transferred to a porcelain crucible in the manner directed at p. 9, melted and weighed.

Volumetrical method.

The readiness with which the chloride of silver subsides in a liquid from which it is precipitated, admits of the point of perfect precipitation being observed with great delicacy; and this method of estimation has superseded all others, on account of its simplicity and accuracy.

The reagent used is chloride of sodium, and the reaction, supposing the silver to be dissolved in the state of nitrate,—

$$AgO,NO_5 + NaCl = AgCl + NaO,NO_5.$$

The normal solution may be made by dissolving 5·86 grms. of chloride of sodium in water to the volume of one litre. A cubic centimeter of this solution (containing 0·00586 grm. NaCl) will be equivalent to 0·01 grm. of silver.

In a great number of instances this normal solution may be used for estimating silver in a solution by simply observing the volume required for perfect precipitation, and then multiplying 0·01 by the number of cubic centimeters. Thus, if 20·5 cubic centimeters are used, the quantity of silver x will be

$$x = 0{\cdot}01 \times 20{\cdot}5 = 0{\cdot}205.$$

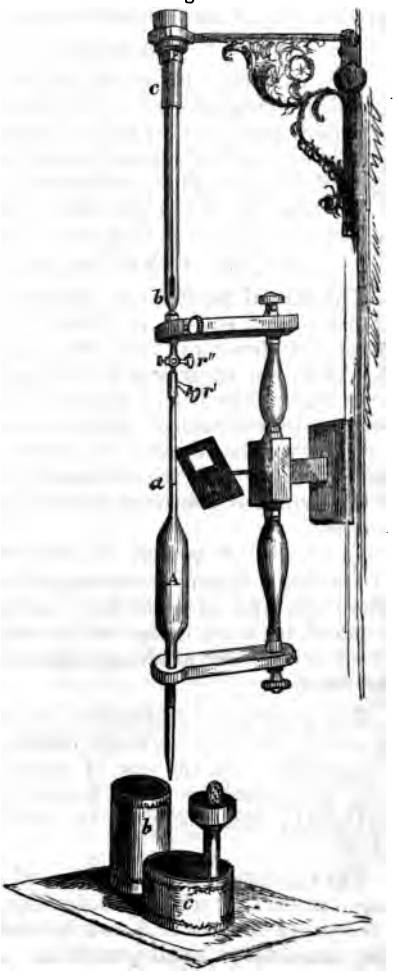
Another solution, containing in one litre a tenth of the above quantity of chloride of sodium (= 0·586 grm.), is required in many instances for estimating small quantities of silver, and for attaining a greater degree of accuracy in the results.

The quantity of material to be taken for an experiment, to obtain direct per-centage results, will depend upon its composition. In the case of nitrate of silver, for instance, 1·70 grm. must be taken to ascertain the per-centage of AgO,NO_5, and 1·08 grm. to ascertain the per-centage of silver.

The experiment. The solution of the substance is introduced into a tolerably capacious stoppered bottle, mixed with a few drops of nitric acid, and the normal solution added from the volumeter in small quantities. After each addition, the

liquid is well shaken and allowed to stand until it becomes clear; a single drop of the solution is then added, and if a further precipitate is produced, the liquid is cleared by shaking, another drop added, and this repeated until a drop of normal solution does not produce any precipitate, or at most a very slight cloud at the surface after a few minutes' rest.

Fig. 43.



In the valuation of alloys containing a tolerably constant proportion of silver, such as silver coin, it is far preferable to take a quantity of the alloy that will contain rather more than 1·0 grm. or 0·5 grm. of silver, and to add at once such a volume of normal solution as will precipitate nearly the whole of the silver, and then to complete the precipitation with the decimal solution.

The apparatus for this purpose consists of a bulbed pipette A (fig. 43), with a narrow neck, and spout with a very small aperture. Upon the neck is a scratch, indicating the required

volume, and the upper end has a collar of caoutchouc tube, by means of which it may be fitted to the delivery-pipe of a large vessel containing the normal solution. This delivery-pipe is a siphon, upon the longer limb of which is fitted a platinum cap, closed by a stopcock r'', with a narrow spout 3 inches long. To admit of the escape of air while the pipette is being filled from above, the spout of the cock r'' is enclosed in a wider tube screwed to the lower side of the cock, and furnished with a lateral valve-cock r' at about 2 inches above the end of the spout of r''. Upon this tube the pipette is fitted by its caoutchouc collar, and when it is to be filled, the fore-finger is placed firmly against the beak, and the cocks r' and r'' opened successively. When the solution has risen just above the scratch on the neck of the pipette, the cocks are both closed in the reverse order, the finger removed, the cock r' very slightly opened, so as to let the solution run out of the pipette until it reaches the mark on the neck, when it is closed, the beak of the pipette wiped with a sponge, and the solution run off into the bottle containing the solution of silver by opening the cock r'. A thermometer may be fitted in the longer limb of the siphon to indicate the temperature of the solution.

In order to save time, and for the sake of convenience when a considerable number of silver estimations are to be made, the sponge is fixed upon a support, together with a receptacle *c*, for the waste solution, and another *b*, for the bottle containing the silver solution, arranged to slide backwards and forwards to such a distance, that the sponge or the mouth of the bottle is brought exactly under the beak of the pipette.

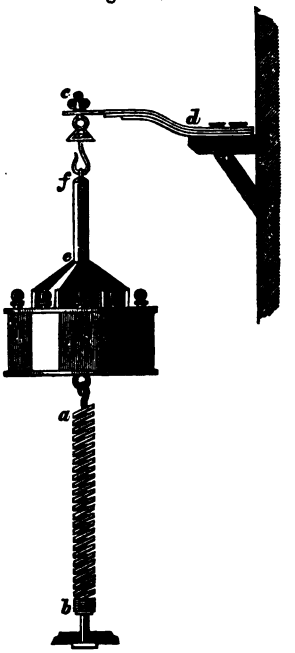
When any number of experiments are to be made, this plan is the only one that can be adopted, because it is much quicker than the other, and affords less chance of error.

Fig. 44.

The experiments are made in series of ten; the weighed quantities of alloy are introduced into bottles, numbered from 1 to 10, arranged in a stand (fig. 44), 5 or 6 grms. of nitric acid added to each by means of a pipette measure, and the stand, together with the bottles, placed in hot water, in order to facilitate solution.

When the metal is completely dissolved, the bottles are allowed to cool, and the nitrous vapour displaced by blowing into them through a piece of bent tube. 100 cubic centimeters of normal solution are then measured into each bottle, the stoppers put in, and the whole transferred to another stand, suspended between two steel springs, so that the ten bottles may be shaken at the same time (fig. 45).

Fig. 45.



When the liquids are clear, the bottles are ranged upon a black shelf with ten compartments, numbered to correspond with the bottles, the stoppers hung upon wires attached to each compartment, and a cubic centimeter of decimal solution added to each bottle by means of a small tube-pipette passing through the cork of the decimal solution bottle (fig. 46).

For each of the bottles in which a precipitate is formed by the decimal solution, a mark is made on its compartment of the shelf; the bottles are again shaken, and the same operation repeated until no further precipitate is formed on the addition of a cubic centimeter of decimal solution.

Fig. 46.

Then supposing that 100 cubic centimeters of the normal solution at 60° F. precipitates exactly 1·0 grm. of silver, in the state of chloride, the quantity of silver in each instance will be found by adding to 1000 the number of cubic centimeters of decimal solution required to complete the precipitation. If, for instance, a certain quantity q of an alloy requires over and above

the 100 cubic centimeters of normal solution, 5 cubic centimeters of decimal solution, the quantity of silver in it will be 1·005 grm.

The per-centage of silver x' is then found by a simple proportion,

$$x' : 1000 = x : q;$$

and when, as in the valuation of alloys that are uniform in composition, q is a constant quantity containing as a minimum 1·0 grm. of silver, a table may be constructed, in which the value in thousandths is indicated by reference to numbers corresponding with the number of cubic centimeters of decimal solution used in each instance.

The quantity of alloy q will, of course, vary according to its value in silver, and the tables likewise will refer only to particular standards. In this country the standard of silver coin is $\frac{925}{1000}$, and the following table expresses the value of the alloy within the limits of variation:—

Quantity of alloy for experiment 1·085 grm.

	0	1	2	3	4	5	6
0	921·6	922·6	923·5	924·4	925·3	926·2	927·2
0·25	921·9	922·8	923·7	924·6	925·6	926·5	927·4
0·50	922·1	923·0	923·9	924·9	925·8	926·7	927·6
0·75	922·3	923·6	924·2	925·1	926·0	926·9	927·9

The numbers in the first column express fractions of thousands, which it is quite possible to estimate after some practice, from the relative thickness of the last precipitate produced by the decimal solution. In the above example, the value of the alloy will be found at the top of the column under 5, as containing $\frac{926·2}{1000}$; or if the fifth cubic centimeter of decimal solution gave a precipitate equal only to ·0005, the value will be found at the third line of the column under 4, corresponding to 4·50, as $\frac{925·8}{1000}$.

It is difficult to ensure the constant equivalence of 100 cubic centimeters of chloride solution to 1 grm. of silver, by reason both of evaporation and change of temperature, and it is ad-

visable never to make the attempt, because accurate results may be obtained with greater certainty otherwise.

With each set of experiments the exact value of the solution for the time is estimated by means of pure silver, of which 1·001 grm. is dissolved and treated with 100 cubic centimeters of normal solution in the usual way. If that volume is exactly equivalent to 1·0 grm. of silver, a precipitate will be produced by the first cubic centimeter of decimal solution, but none by the second. But if a second and third cubic centimeter of decimal solution are required to complete the precipitation in this proof experiment, then the 100 cubic centimeters will be equivalent to only 1·001 − ·003 = ·998, so that to ascertain the amount of silver in the several experiments, it will be necessary to deduct from the number of cubic centimeters of decimal solution required for perfect precipitation, the number required, by the 1·0 grm. of pure silver. In the present instance this correction is 2, and the result of an experiment, in which 5 cubic centimeters of decimal solution was required, would be 1·003 grm. of silver.

Other metals do not by their presence influence the results obtained by this process, except mercury, which is precipitated in the state of chloride. However, when the proportion of mercury is at all considerable, it is easily recognized by the difficulty of rendering the liquid clear by agitation, and by the precipitate of chloride remaining white. In such a case the inaccuracy of the results may be obviated by adding to the nitric solution of silver some acetate of soda, which prevents the precipitation of the mercury.

The approximative estimation of the value of the alloy, necessary as a preliminary to this process, may be made either by cupellation with lead, or by the ordinary volumetrical experiment with a burette.

QUALITATIVE ANALYSIS OF ALLOYS OR METALS SUSPECTED TO CONTAIN IMPURITIES.

For the qualitative examination of metallic substances, the following systematic treatment should be adopted.

The substance is treated with nitric acid in a glass flask, and the reaction assisted by the application of heat.

In almost all probable cases the result will come under one of the following heads.

I. A reaction takes place, red nitrous fumes being evolved; and in this case—

A. The solution of the substance takes place without any residue, indicating the absence of *gold* (*platinum* ?), *antimony*, and *tin*.

a. The solution may contain all the other metals likely to be present in alloys, &c.; *copper*, *lead*, *silver*, *bismuth*, *mercury*, *arsenic*, *zinc*, *nickel*, *cobalt*, *iron*.

B. The solution is partial, and the residue is

b. *metallic*, or a *black powder*, indicating the presence of *gold*, *platinum*, and perhaps *antimony* and *tin* in small proportions.

β. *white*, indicating the presence of *antimony* or *tin*, and perhaps *gold* and *platinum* in small proportions.

C. No solution takes place, indicating the absence of all metals but *gold*, *platinum*, *antimony*, *tin*, and perhaps *silver* and *lead* in small proportions. The residue may be

c. *white*, indicating the same as *β*; or:—

II. No reaction takes place, and

D. The substance remains unaltered, indicating the probable absence of all metals but *gold* and *platinum*.

The solution *a* is in all cases treated in the same manner. When a residue remains, it is separated by decantation or by filtration, after the excess of acid has been removed by boiling. The metals are tested for in the following manner, or according to the system described in Chapter X.

1. Copper. When in any considerable proportion, the blue colour of the solution indicates its presence; when in small quantity, it is indicated by the production of an intense and characteristic blue colour on the addition of an excess of ammonia, the reactions of any other metals that may be present in the solution being disregarded.

2. Lead is indicated by the formation of a white precipitate of sulphate of lead on the addition of a few drops of dilute sulphuric acid.

This indication may be confirmed by collecting the precipitate and fusing it with carbonate of soda on charcoal before the blowpipe. The sulphate is reduced, yielding soft beads of metal and a yellow incrustation upon the charcoal.

3. **Silver** is indicated by the formation of a white precipitate (AgCl) on the addition of hydrochloric acid. When the alloy contains lead, this test should be applied to the liquid filtered from the sulphate of lead in the previous experiment, because, as chloride of lead is precipitated under the same circumstances, it might lead to a false result. Chloride of lead and chloride of silver may, however, be distinguished by their behaviour with ammonia; the former is unaltered, while the latter is dissolved by this reagent.

4. **Bismuth** is indicated by the formation of a white precipitate when the nitric acid solution, evaporated, not to dryness, but so as to remove excess of acid, is poured into water.

When the experiment 3 has shown the presence of silver, the presence of bismuth is improbable; and this is likewise the case when the solution of the alloy by nitric acid is complete and 1 has indicated the presence of copper, for alloys containing copper and free from tin or antimony, as well as alloys of silver, in which there is an intentional admixture of bismuth, are not known. In these instances therefore the application of the test 4 may be omitted.

As a confirmation of the above reaction, the precipitate is fused on charcoal by means of the blowpipe. It should become yellow, and on the addition of carbonate of soda yield reddish-white brittle beads of metal.

5. **Mercury.** When a fragment of the metal heated in a dry test-tube yields a sublimate, it may contain either mercury or arsenic. (Likewise antimony, which would have been already indicated by the treatment with nitric acid.) The mercurial sublimate is in the form of a gray film, which, when rubbed with a roll of paper, collects into metallic globules.

The presence of mercury is confirmed by the reduction of its salts in contact with copper, and the formation of an amalgam on the surface of the latter. For this purpose a few drops of the nitric acid solution are freed from excess of acid by heating in a porcelain capsule, a little water added,

and a piece of bright copper laid in the liquid. When mercury is present it becomes grayish, and when rubbed with the finger takes a white metallic lustre.

6. Arsenic is indicated when the sublimate obtained, as in the last experiment, forms upon the glass a blackish brilliant film, the appearance of which is preceded by the evolution of white vapour and the odour of garlic peculiar to this metal.

The detection of arsenic in small quantity is best effected by means of Marsh's apparatus, into which a few drops of the nitric acid solution are introduced for the purpose, and the experiment conducted with the precautions and in the manner described at p. 129.

In order to confirm these indications of arsenic, the porcelain plate, upon which the films have been produced, is laid over a capsule containing sticks of phosphorus half-immersed in water. When the films are arsenical they disappear in a few hours, while those of antimony remain for a much longer time unaltered.

The remarks made above as to the occurrence of bismuth in alloys, apply equally to arsenic.

7. Zinc. Indicated by the formation of a white precipitate (ZnS) on treatment with alkaline sulphurets.

When the solution of the alloy has given indications of any of the above metals, whose coloured sulphurets would disguise the zinc reaction, they must be separated, before testing, by sulphuretted hydrogen, and the filtered liquid boiled with a few drops of nitric acid to remove the excess of this gas. When zinc is present the addition of caustic soda produces a white precipitate, which dissolves in excess of the reagent, and from this solution sulphuretted hydrogen throws down zinc in the form of a white precipitate (ZnS).

As a confirmatory test, the precipitate of oxide is moistened with cobalt solution and heated by means of the blowpipe. Under these circumstances the mass acquires a green colour, owing to the formation of a compound of oxide of zinc and protoxide of cobalt.

8. Nickel. Indicated by the formation of an apple-green precipitate (NiO,HO) on the addition of caustic soda. The oxide of nickel does not, like oxide of zinc, dissolve in caustic soda; and as zinc and nickel may occur together in an alloy,

it is always necessary, when a green precipitate is obtained by treatment with caustic soda, as in this and the last experiment, to employ an excess of the reagent and to test the alkaline solution for zinc by sulphuretted hydrogen.

9. Iron. Indicated when the precipitate produced by caustic soda, and not dissolved by an excess of that reagent, has a dirty green or brown colour, instead of a bright apple-green.

This precipitate is dissolved in hydrochloric acid, carbonate of soda added to the liquid, drop by drop, until it becomes reddish-brown. Some acetate of soda is then added and the whole boiled. Iron is precipitated as hydrated peroxide (Fe_2O_3,HO); nickel, when present, remains in solution, and may be tested for as above with caustic soda.

There is little probability that iron would be intentionally present in an alloy of copper free from tin, which would be perfectly soluble in nitric acid. It may, however, occur accidentally, but then only in very small proportion.

When on treatment with nitric acid there is produced an insoluble residue, β, it is washed upon a filter and boiled with tartaric acid in a flask.

10. Tin. Indicated by the insolubility of the oxide in solution of tartaric acid.

As a confirmatory test, the powder undissolved by tartaric acid is fused with carbonate of soda on charcoal by means of the blowpipe. The oxide of tin is reduced, yielding malleable globules of metal.

11. Antimony. Indicated by the solubility of the oxide in boiling solution of tartaric acid.

As a confirmation, the solution is evaporated to dryness and the residue heated upon charcoal by means of the blowpipe. The oxide of antimony is reduced, yielding brittle metallic globules, which burn and give off a thick white vapour of oxide of antimony.

When the substance is not acted upon by nitric acid, and when it is but partially dissolved, the residue, *b*, left is treated with a mixture of 3 parts hydrochloric acid and 1 part nitric acid with heat.

The result of this treatment may be either—

a. *Partial solution and the separation of a white insoluble powder, chloride of silver, or* (less probable) *chloride of lead.*

Although silver is soluble in nitric acid and therefore belongs to the previous group, it possesses the peculiarity of being insoluble when alloyed in certain proportions with gold and some other metals.

b. *Complete solution.*

In both the above cases the metals belonging to this group will be in solution.

12. Gold. Indicated by the formation of a brown precipitate of metallic gold on the addition of protosulphate of iron. The precipitate, collected on a filter and rubbed when dry with some hard substance, acquires metallic lustre. When the proportion of gold in solution is very small, the gold remains for a long time suspended.

It is essential for the success of this experiment, especially when the quantity of gold is small, that the liquid should be freed from nitric acid before the test is applied, for the intense colour produced by the reaction of nitric acid and protosalts of iron would render the gold reaction less perceptible.

13. Platinum. Indicated by the formation of a yellow precipitate ($PtCl_2,NH_4Cl$) on the addition of chloride of ammonium to the chloride solution.

As platinum is not reduced by sulphate of iron, the liquid which has been tested for gold, or from which gold has been separated, may be employed.

The liquid from which gold or platinum has been separated may contain metals, which are only partially dissolved by nitric acid, when alloyed with either of these. Their presence in the alloy would be already indicated in the examination of the nitric acid solution as above.

The chloride solution may moreover contain metals which give rise to the formation of an insoluble white residue when treated with nitric acid—antimony and tin. Their occurrence is unfrequent.

A. COPPER ALLOYS.

a. Consisting of two metals.

I. Copper and tin.	COPPER.	TIN.
1. Chrysocole	95·00	5·00
2. [1]Bell-metal	80·00	20·00
3. „ „	60·00	40·00
4. Gun-metal	90·90	9·10
5. Chinese gong-gong	78·00	22·00
6. Clock bell-metal	75·19	24·81
7. [2]Metal for the sockets of axle-trees for locomotives	86·03	13·97
8. [3]Medal bronze	92·00	8·00
9. „ „ which resists atmospheric oxidation	94·17	5·83

[1] See 72.—[2] See 29, 30, 31, 32, 34, 73, 81, 82.—[3] Sometimes with a little lead.

II. Copper and zinc.	COPPER.	ZINC.
10. Brass	73·00	27·00
	65·00	35·00
„ average	70·00	30·00
11. Pinchbeck	85·00	15·00
12. Princes metal	75·00	25·00
13. Bath metal	55·00	45·00
14. [1]Plating for buttons	43·00	57·00
15. Imitation gold	82·85	17·15
16. Mosaic gold	64·51	35·49
17. [2]Brass solder	61·25	38·75
18. Malleable brass	70·10	29·90
„ „ (superior)	60·00	40·00
19. Brass solder (yellow and easily fusible)	45·00	55·00

[1] See 26 and 27.—[2] See 19 and 33.

III. Copper and silver.	COPPER.	SILVER.
The alloys used for coin and silverware, varying in value according to the several standards legalized in different countries.		
20. English coin and plate	7·50	92·50
21. French „	10·00	90·00
22. American „	10·00	90·00

IV. Copper and gold.	COPPER.	GOLD.
23. Red gold	66·67	33·33

V. Copper and mercury.	COPPER.	MERCURY.
24. Dentists' copper amalgam	30·00	70·00

b. Alloys consisting of three metals.

VI. Copper, tin, and zinc.	COPPER.	TIN.	ZINC.
25. Manheim gold	79·13	13·04	7·83
26. Button metal	62·22	2·78	35·00
27. [1] „ „	58·94	5·28	35·78
28. Alloy for gun-mountings	80·00	3·00	17·00
29. [2]Metal for sliding levers of locomotives	85·25	12·75	2·00
30. The same, according to Fenton	5·50	14·50	80·00
31. Metal for stuffing-boxes of locomotives	90·06	3·56	6·38
32. „ „ cylinders of locomotives	88·63	2·38	6·99
33. [3]White brass solder	57·41	14·60	27·99
34. Metal for the sockets of axle-trees for locomotives (*French*)	82·00	10·00	8·00
35. Bronze, for statues (*Hoffmann*) (extreme red)	84·42	4·30	11·28
36. „ „	83·05	3·92	13·03
37. „ „	81·06	3·62	15·32
38. „ „	78·09	3·44	18·47
39. „ „	73·58	3·15	23·27
40. „ „	70·36	2·76	26·88
41. „ „ (extreme yellow)	65·95	2·49	31·56

[1] See 14.—[2] See 7, 34, 73, 81, 82.—[3] See 17 and 19.

VII. Copper, zinc, and lead.	COPPER.	ZINC.	LEAD.
42. Alloy for electro-plating	68·20	31·60	0·20

VIII. Copper, zinc, and silver.	COPPER.	ZINC.	SILVER.
43. Silver solder (*hard*)	30·00	12·85	57·15
44. „ „ (*hard*)	23·33	10·00	66·67
45. „ „ (*hard*)	26·66	10·00	63·34
46. „ „ (*soft*)	14·75	8·20	77·05
47. „ „ (*soft*)	22·34	10·48	67·18

IX. Copper, silver, and gold.	COPPER.	SILVER.	GOLD.
48. Gold solder	24·24	27·57	48·19
49. Enamel solder	25·00	7·07	67·93
50. Yellow gold	12·50	37·50	50·00
51. English coin	8·34		91·66
52. French „	10·00		90·00
53. American „	10·00		90·00

X. Copper, tin, and antimony.

		COPPER.	TIN.	ANTIMONY.
54.	[1]Britannia metal	1·85	81·64	16·51
55.	„ „	0·91	89·97	9·12
56.	Metal d'Alger	5·00	95·00	trace
57.	Socket metal (*Dawrence*)	22·22	33·33	44·45

[1] See 75, 79, 89.

XI. Copper, zinc, and nickel.

		COPPER.	ZINC.	NICKEL.
58.	German silver, or argentine	40·62	43·76	15·62
59.	„ „	41·47	26·08	32·45
60.	„ „	55·55	5·55	38·90
61.	„ „	53·40	29·10	17·50
62.	[1]Alfenide	59·60	30·30	10·10

[1] Contains a trace of iron.

XII. Copper, tin, and bismuth.

		COPPER.	TIN.	BISMUTH.
63.	White metal for table bells	2·06	97·31	0·63

XIII. Copper, lead, and antimony.

		COPPER.	LEAD.	ANTIMONY.
64.	Reflector metal	82·18	9·22	8·60

XIV. Copper, tin, and arsenic.

		COPPER.	TIN.	ARSENIC.
65.	Reflector metal	66·22	33·11	0·67
66.	„ „	64·00	32·00	4·00

c. Alloys consisting of four metals.

XV. Copper, tin, zinc, lead.

		COPPER.	TIN.	ZINC.	LEAD.
67.	Metal for gilt wares	78·47	2·87	17·23	1·43
68.		64·43	0·25	32·44	2·86
69.		72·43	1·87	22·75	2·96
70.		70·90	2·00	24·05	3·05
71.	[1]Bronze for statues	78·50	2·90	17·20	1·40
72.	[2]Bell metal (*Thomson's*)	80·00	10·10	5·60	4·30
73.	[3]Socket metal for locomotives (*Stephenson's*)	79·00	8·00	5·00	8·00
74.	Bidery	48·48	6·60	33·80	12·12

[1] See 35.—[2] See 2, 3, 5, 6, and 63.—[3] See 7, 29, 30, 31, 32, 34, 69, and 70.

XVI. Copper, tin, zinc, and antimony.

		COPPER.	TIN.	ZINC.	ANTIMONY.
75.	[1]Britannia metal	0·30	89·70	0·30	9·70

[1] See 54, 55, 79, and 89.

XVII. Copper, tin, zinc, and arsenic.

		COPPER.	TIN.	ZINC.	ARSENIC.
76.	Reflector metal (*Little's*) ...	65·01	30·82	2·24	1·83

XVIII. Copper, tin, antimony, and bismuth.

		COPPER.	TIN.	ANTIMONY.	BISMUTH.
77.	Pewter	6·77	84·74	6·77	1·72
78.	„	1·79	89·28	7·14	1·79
79.	[1]Britannia metal.....	1·78	89·30	7·14	1·78

[1] See 54, 55, 75, 89.

XIX. Copper, zinc, silver, gold.

		COPPER.	ZINC.	SILVER.	GOLD.
80.	Gold solder	26·25	6·25	31·25	36·25

XX. Copper, tin, zinc, and iron.

		COPPER.	TIN.	ZINC.	IRON.
81.	Socket metal for locomotives (*Belgian*).....	89·02	2·44	7·76	0·78

d. Alloys consisting of five metals.

XXI. Copper, tin, zinc, lead, and iron.

		COPPER.	TIN.	ZINC.	LEAD.	IRON.
82.	Socket metal for locomotives... (*English*)	73·96	9·49	9·03	7·09	0·43

XXII. Copper, tin, zinc, arsenic, and platinum.

		COPPER.	TIN.	ZINC.	ARSENIC.	PLATINUM.
83.	Reflector metal (*Cooper's*).....	57·86	27·28	3·30	1·65	9·91

B. ALLOYS WITHOUT COPPER.

a. Alloys consisting of two metals.

XXIII. Tin and zinc.

		TIN.	ZINC.
84.	Spurious silver leaf	90·09	9·91

XXIV. Tin and lead.

		TIN.	LEAD.
85.	Solder	50·00	50·00
86.	„	66·67	33·33
87.	„	33·33	66·67
88.	Silver solder	63·00	37·00

XXV. Tin and antimony.

		TIN.	ANTIMONY.
89.	[1]Britannia metal	90·00	10·00

[1] See 54, 55, 75, 79.

XXVI. Tin and mercury.

		TIN.	MERCURY.
90.	Amalgam for mirrors	70·00	30·00
91.	[1] „ „ curved mirrors	80·00	20·00

[1] See 112.

XXVII. Lead and antimony.

		LEAD.	ANTIMONY.
92.	Type-founders' metal	9·91	90·09
93.	„ „ „	20·00	80·00

and a great number of other proportions.

XXVIII. Lead and arsenic.

		LEAD.	ARSENIC.
94.	Shot metal	97·07	2·93
95.	„ „	99·60	0·40

XXIX. Zinc and silver.

		ZINC.	SILVER.
96.	Reflector metal (*Doppler's*)	20·00	80·00

XXX. Silver and gold.

		SILVER.	GOLD.
97.	Green gold	25·00	75·00
98.	Yellow gold	66·67	33·33
99.	[1]Enamel solder	19·57	80·43

[1] See 40.

XXXI. Bismuth and mercury.

		BISMUTH.	MERCURY.
100.	Amalgam for coating spherical mirrors	80·00	20·00

XXXII. Gold and steel.

		GOLD.	IRON.
101.	„ „	80·00	20·00
102.	„ „	83·33	16·67

b. Alloys consisting of three metals.

XXXIII. Tin, lead, and bismuth.

	TIN.	LEAD.	BISMUTH.
103. Fusible metal (*Newton's*)	20·03	29·97	50·00
104. „ „ (*Rose's*)	25·00	25·00	50·00
105. Bismuth solder	33·33	33·33	33·34
106. Metal for calico-printing blocks ...	50·00	33·34	16·66
107. [1]Metal for casting Perrotine blocks...	33·33	33·34	33·33

[1] See 115.

XXXIV. Tin, zinc, and mercury.

	TIN.	ZINC.	MERCURY.
108. Amalgam for electrical machines..... (*Kienmayer*)	25·00	25·00	50·00
109. Amalgam for electrical machines..... (*Singer*) ...	11·11	22·22	66·67

XXXV. Silver, gold, steel.

	SILVER.	GOLD.	IRON.
110. Gray gold	5·89	88·23	5·88

XXXVI. Silver, platinum, gold.

	SILVER.	PLATINUM.	GOLD.
111. [1]Dentists' gold.....	8·34	66·67	24·99

[1] Sometimes in other proportions.

c. Alloys consisting of four metals.

XXXVII. Tin, lead, bismuth, and mercury.

	TIN.	LEAD.	BISMUTH.	MERCURY.
112. [1]Amalgam for curved mirrors.....	8·33	8·34	8·33	75·00
113. Metal for anatomical injections	16·41	9·27	27·81	46·51

[1] See 91.

XXXVIII. Tin, lead, bismuth, and antimony.

	TIN.	LEAD.	BISMUTH.	ANTIMONY.
114. Queen's metal.....	75·00	8·33	8·34	8·33
115. [1]Metal for casting Perrotine blocks	48·00	32·50	9·00	10·50

[1] See 107.

The unalloyed metals employed for technical purposes are rarely quite free from admixtures—generally small in amount—of other metals or of non-metallic substances, originating either from the ores or the materials used in smelting. Some of these substances are always very prejudicial to the utility of the metals, others only when they exceed a certain proportion, and it is frequently desirable to have recourse to chemical tests of purity.

The examination of metals for these impurities is conducted in the same manner as the qualitative analysis of alloys, and for the convenience of the analyst, the substances with which they are respectively likely to be contaminated are enumerated below, with references to the methods of testing for them (p. 159), and descriptions of the tests to be applied in cases which are not considered in the qualitative analysis of alloys.

Zinc may contain:—

Tin..... } Tested for in the residue β (p. 162).
Antimony }

Copper .. } Tested for in the nitrate solution *a*, 1 and
Lead .... } 2 (p. 159).

Arsenic .... Tested for as in 6 (p. 161).

Cadmium.... Frequently associated with zinc. It is tested for in the precipitate produced by sulphuretted hydrogen. When this is dissolved by nitric acid, the solution separated from sulphur by filtration, mixed with excess of carbonate of ammonia, and allowed to remain for some time in a covered vessel, the cadmium—perhaps with lead and bismuth—is obtained as a precipitate. It may be obtained separately by dissolving the precipitate by hydrochloric acid, and adding excess of ammonia, which dissolves oxide of cadmium, but not the oxides of lead or bismuth.

Iron .... } Tested for as in 8 and 9 (p. 161 and 162).
Nickel .. }

Cobalt .. *Manganese* — These metals react with caustic soda in the same manner as iron and nickel, and would therefore be contained in the precipitate produced by that reagent as in 8 (p. 161). They may be separated as directed at p. 128.

Sulphur .... Tested for in the nitrate solution by chloride of barium, as it would be converted into sulphuric acid by treatment with nitric acid.

Iron may contain:—

Copper .... Tested for in the solution *a*, 1 (p. 159).

Arsenic .... Tested for as in 6 (p. 161).

Manganese.. Tested for in the precipitate produced by caustic soda. It is dissolved in nitro-hydrochloric acid, the iron precipitated as succinate by adding to the solution, mixed with ammonia until a very slight permanent precipitate is produced, a *neutral* solution of succinate of ammonia, as long as any precipitate is produced. The manganese is then precipitated from the filtrate by sulphide of ammonium, washed, dissolved by hydrochloric acid, and precipitated in the state of carbonate for estimation.

Sulphur .... Tested for in the nitric solution with chloride of barium, and estimated as sulphate of baryta in the manner described at p. 78.

Carbon .... Always present in iron of all kinds. When the metal is dissolved in weak hydrochloric acid, a portion of the carbon is converted into gaseous carburetted hydrogen, which may be recognized by the odour. Another portion remains as an insoluble black residue. This latter carbon exists in the iron in an uncombined state as graphite, while the former is combined with iron as a true carburet.

The uncombined carbon is estimated by collecting upon a dry weighed filter the residue, consisting of carbon and silica, left on solution, washing it with water, then with a little æther, and weighing the whole after desiccation at 212° F. The filter with its contents is then ignited in a platinum crucible until the whole of the carbon is burnt, when the residue is weighed, and the difference between the two weights gives the amount of carbon.

The total amount of carbon is estimated in the manner described at p. 194.

Silicium.... Tested for in the nitric solution, by evaporating to dryness at 212° F., and digesting the residue with weak hydrochloric acid, when it remains as a white insoluble powder of silica.

Care must be taken to prevent any error in this estimation that might arise from the presence of particles of slag, which are not unfrequently disseminated through the mass of metal. When this is the case, it should be treated with very weak hydrochloric acid, which dissolves the iron without seriously attacking the slag.

Phosphorus.. Tested for in the solution from which silica has been separated as above, and iron and manganese, by digesting with an excess of sulphide of potassium at 212° F. for several hours. The phosphorus will then be in solution as phosphoric acid; it may be separated from the iron by filtration, and estimated as directed at p. 178.

Nickel may contain:—

Copper.... } Tested for in the nitric acid solution.
Lead .... }

Arsenic .... Tested for by Marsh's test, or as in 6 (p. 161).

Iron ....
Cobalt ..
Manganese } Tested for in the precipitate from 8, p. 161.

Carbon ..
Sulphur .. } Tested for as in iron and zinc.

Tin may contain:—

Bismuth..
Copper ..
Lead ....
Iron ....
Zinc .... } Tested for in the nitric acid solution.

Antimony .. Tested for in the residue β, as in 11.
Arsenic.... Detected by Marsh's test.

Tin-foil is immediately and completely converted into a white powder when treated with moderately dilute nitric acid, and it may thus be distinguished from lead-foil coated with tin, which by the same treatment leaves the lead unacted upon.

Antimony may contain:—

Sulphur.... Tested for in the nitric acid solution.
Arsenic.... Tested for as in 6 (p. 161).

Gold may contain:—

Silver.... { Indicated on solution in nitrohydrochloric acid, by a white residue, AgCl.
Copper .... Tested for in the solution *a*.
Platinum .. Tested for as in 13, p. 163.

Gilt surfaces may be tested by treatment at different spots with metallic mercury and subnitrate of mercury. When a globule of mercury is rubbed with a piece of kid leather upon the surface of true gilding, a white silvery spot is immediately produced, while imitation gilding suffers no alteration of colour. But when a drop of subnitrate of mercury is rubbed upon the surface, the latter—containing copper and tin—becomes white in a few minutes, while true gilding remains unaltered. Varnish or grease must be removed by means of alcohol before these tests are applied.

Electro-gilding may be distinguished from fire gilding by

dissolving the underlying metal in moderately strong nitric acid, so as to separate the gold in small laminæ, which in the case of electro-gilt surfaces are of the same colour and brightness on both sides, while in the case of fire gilding, the under surface next to the metal basis is darker, dead and brownish. Further, the laminæ from fire gilding present fine pores when held against the light, while those from electro-gilding appear continuous.

Copper may contain:—

Tin, *Antimony* — Tested for in the residue β.

Bismuth, *Lead*, *Iron*, *Zinc*, *Arsenic*, *Sulphur*, *Carbon* — Tested for in the solution *a*.

Lead may contain:—

Antimony, *Gold* — Tested for in the residue β.

Copper, *Silver*, *Zinc* — Tested for in the solution *a*.

Arsenic .... Tested for as in 6 (p. 161).

Mercury may contain:—

Lead and other metals, which, when not volatile, may be easily detected by distilling a small portion in a glass retort. The residue is tested in the usual manner.

Silver may contain:—

Copper .... Indicated by the colour of the nitric solution, and when in small proportion by a brown precipitate with ferrocyanide of potassium after the separation of the silver by hydrochloric acid.

Gold ..... Frequently present in small proportion. Remains undissolved by nitric acid as a black sediment.

Imitations of silver may be distinguished from the ordinary alloys of silver and copper by the following test:—The metal to be examined is immersed in a mixture of bichromate of potash (0·6 grm.) with sulphuric acid (1·0 grm.) and water (8 grms.), by the action of which silver acquires a red colour, which is darker the larger the proportion of silver. Or:—

The metal is rubbed upon a piece of slate, the streak wetted with nitric acid, by which it is dissolved, and then a drop of hydrochloric acid added by a glass rod; when a curdy precipitate is produced, or, in the case of very small proportions of silver, only an oily glitter at the surface of the liquid, the presence of silver is certain. The precipitate of chloride of lead that might in some instances be formed, is readily distinguishable from the chloride of silver by its disappearance on the addition of a larger quantity of water.

CHAPTER VIII.

NON-METALLIC ELEMENTS AND THEIR COMPOUNDS KNOWN IN COMMERCE; THEIR CHARACTERS, THE TESTS OF THEIR PURITY, AND THE METHODS OF VALUATION.

Sulphur, S. 16.

This substance occurs in a crude state, in the form of rolls; as a yellow powder; and, for medical purposes, as a very fine white powder. The probable impurities are,—

Earthy substances, indicated by a residue left on sublimation. Precipitated sulphur is sometimes largely adulterated with burnt gypsum or sulphate of lime.

Arsenic, tested for by digesting the finely powdered sulphur in caustic ammonia for some time, and indicated in the filtrate by a yellow precipitate of sulphuret of arsenic on the addition of hydrochloric acid. When the quantity is very small, the hydrochloric acid may not produce a precipitate immediately, until the filtrate, mixed with a few drops of potash solution, is evaporated to a small bulk.

Sulphuric acid, in small proportion, owing to a gradual oxidation by the atmosphere.

The reactions presented by sulphur vary according to its state of combination. The compounds of sulphur with metals (*sulphides*) are for the most part insoluble, and when heated evolve the peculiar odour of burning sulphur, owing to the formation of sulphurous acid. Many of these sulphides, and all those which are soluble, evolve sulphuretted hydrogen when treated with dilute sulphuric acid.

The sulphur of sulphides is generally estimated as sulphuric acid—into which it is converted by means of strong nitric acid or nitrohydrochloric acid—either by weighing the sulphate of baryta obtained, or by the volumetrical method (p. 78).

When in the state of sulphide of hydrogen or sulphurous acid, sulphur, or these compounds themselves, may be esti-

mated by the volumetrical method with bichromate of potash in the same manner as tin (p. 133), the reactions in each case being—

1. $2(Cr^2O^3) + 3HS = Cr^2O^3 + 3S + 3HO.$
2. $2(CrO^3) + 3SO^2 = Cr^2O^3,3SO^3.$

The end of the reaction is indicated, as in the estimation of tin, by means of iodide of potassium and starch; and when the sulphuretted hydrogen or sulphurous acid to be estimated is in small proportion, as in mineral water, &c., the normal solution of bichromate may be diluted so as to furnish more accurate results.

Phosphorus, P. 31.

Colourless, or slightly yellow, translucent, solid. It fuses at 111° F. to a transparent oily liquid.

The so-called *amorphous phosphorus* is a dark red powder, which may be heated to 482° F. without melting. At the ordinary temperature it is without odour. The possible impurities are,—

Sulphur, tested for by adding chloride of barium to a solution of the phosphorus in nitric acid.

Arsenic, tested for by sulphuretted hydrogen in the solution from which excess of nitric acid has been removed by boiling.

The reactions indicative of phosphorus vary, as in the case of sulphur, according to its state of combination. When combined with metals as *phosphides*, it is oxidized by nitric or nitro-hydrochloric acid, and dissolved as phosphoric acid. The excess of nitric acid is removed by evaporating the solution to dryness; the residue dissolved in water; the metals separated by sulphuretted hydrogen, or by digestion for some hours with sulphide of potassium, and phosphoric acid tested for in the filtrate.

The distinctive character of phosphoric acid in a free state, and in such of its compounds as are soluble in water, is the formation, with sulphate of magnesia, in the presence of ammonia and chloride of ammonium, of a colourless crystalline precipitate of phosphate of magnesia and ammonia ($2MgO, NH_4O,PO_5$).

Phosphoric acid may be best tested for in compounds insoluble in water, by molybdate of ammonia, which produces

in the cold nitric acid solution of the substance a yellow precipitate, insoluble in dilute acid, but soluble in caustic or carbonated alkalies.

Phosphoric acid is estimated as pyrophosphate of magnesia ($2MgO,PO_5$), by igniting the precipitate of phosphate of ammonia and magnesia.

Phosphoric acid may also be estimated indirectly by the volumetrical method, when alumina is absent. The process is based upon the facts that—

1. Phosphoric acid (tribasic) is precipitated by neutral acetate of iron ($Fe_2O_3 3\bar{A}$) from solutions containing no free acid but acetic, as phosphate of iron (Fe_2O_3,PO_5); and

2. That the phosphates insoluble in water are, with the exception of the phosphates of iron and alumina, dissolved by acetic acid.

Therefore, by estimating the amount of iron *fe* in the precipitate of phosphate of iron obtained, the quantity of phosphoric acid *x* may be calculated by the proportion—

$$x : fe = PO_5 : Fe_2.$$

The iron is estimated, after reduction to the state of protoxide, by the normal solution of permanganate of potash diluted to five times its volume, so that 100 cubic centimeters are equivalent to 0·560 grm. of iron or 0·713 grm. phosphoric acid. The quantity of the phosphoric acid may be found by multiplying 0·00713 by the number of cubic centimeters of permanganate solution required.

The quantity of material to be taken for experiment is 0·713 grm., and then the number of cubic centimeters of solution indicate directly the per-centage of phosphoric acid.

The experiment. To obtain a solution fit for the precipitation of the phosphoric acid, phosphates soluble in water are mixed with a tolerably large excess of acetic acid; those insoluble in water are dissolved in hydrochloric acid, and the liquid mixed with sufficient acetate of soda to neutralize all the hydrochloric acid.

This solution is then mixed with neutral acetate of iron ($Fe_2O_3,3PO_5$) until there is no further precipitation and the liquid acquires a brownish-red colour. The yellowish-white precipitate is collected upon a moistened filter washed with *cold* water until all the excess of iron is removed, and then

dissolved in hydrochloric acid, the iron reduced to the state of protoxide by means of zinc (see p. 124), and lastly, the iron estimated by permanganate solution.

The reagent ($Fe_2O_3,3\bar{A}$) for precipitating the phosphate of iron is liable to decomposition when kept, and it is therefore advisable to prepare it fresh for each experiment by mixing together equal volumes of solutions containing respectively in a litre 48 grms. of double sulphate of iron and ammonia ($Fe_2O_3 3SO_3 + NH_4O,SO_3 + 24$ aq.) and 40·86 grms. of crystallized acetate of soda ($NaO,A + 6$ aq.) and kept ready for the purpose.

Compounds of phosphoric acid with peroxide of iron are weighed in a dry state, dissolved in hydrochloric acid, the iron reduced by zinc, and estimated with the permanganate solution. The quantity of phosphoric acid is estimated by deducting from the total quantity of substance experimented upon, the quantity of oxide of iron (Fe_2O_3) equivalent to the iron found.

For the estimation of phosphoric acid in phosphate of alumina, which, like phosphate of iron, is insoluble in acetic acid, the substance is melted with silica and carbonate of soda. By this treatment there are formed silicate of alumina and phosphate of soda: the latter is separated by water and the solution treated as above directed (p. 178). Or when the melted mass is covered with nitric acid, mixed with metallic mercury, and heated until all the nitric acid is neutralized, the phosphoric acid is precipitated as phosphate of suboxide of mercury, which may be heated with carbonate of soda and the acid estimated in the solution.

The bibasic modification of phosphoric acid may be estimated in a similar manner, the precipitation of the acid being effected by iron alum and not with acetate of iron. To obtain direct per-centage results, the quantity of material to be taken for experiment is 0·4284 grm., and the permanganate solution must be diluted to ten times its volume, so that 100 cubic centimeters are equivalent to 0·280 grm. of iron.

Phosphoric acid may be estimated in urine by means of a normal solution of chloride of iron (Fe_2Cl_3), as free as possible from excess of acid. It is prepared by dissolving 15·556 grms. of pure iron in hydrochloric acid, adding enough nitric acid

to convert the whole into chloride, then evaporating to dryness, and dissolving the residue in water to the volume of one litre. A cubic centimeter of this solution is equivalent to 0·010 grm. of phosphoric acid.

The experiment is made with 100 cubic centimeters of urine mixed with acetic acid, and the normal solution added until a drop of the urine produces a blue colour in paper saturated with ferrocyanide of potassium. This test is applied by letting the drop fall upon a piece of filter paper placed above the test paper, and thus doing away with the necessity for filtration.

Chlorine, Cl. 35·5.

This substance may be detected in a free state by the liberation of iodine from iodide of potassium. Paper saturated with a solution of this salt and starch, immediately becomes blue, when moistened and brought in contact with free chlorine.

The distinctive character of chlorine, combined with metals as *chlorides*, is the formation with nitrate of silver of a white curdy precipitate (AgCl), insoluble in nitric acid and soluble in ammonia. In testing for chlorine in substances insoluble in water, they must first be fused with an excess of carbonated alkali, and the filtered solution, mixed with excess of nitric acid, tested with nitrate of silver.

Chlorine, in a free state, is estimated by digesting the aqueous solution with a known quantity of subchloride of mercury until the smell disappears. The subchloride is converted into soluble chloride, and the amount of chlorine x may be calculated from the difference y in the weight of the subchloride before and after the experiment by the proportion—

$$x : y = \mathrm{Cl} : \mathrm{Hg^2Cl}.$$

Chlorine, in combination with metals, may be estimated by precipitation as chloride of silver, either by weighing the precipitate, or by means of a normal solution of silver, in a similar manner to the estimation of silver. In the latter case, the end of the reaction may be rendered more easily ascertainable by adding to the solution of chloride a few drops of phosphate of soda solution. So long as any chlorine remains in solution, there is no *permanent* formation of phos-

phate of silver, but as soon as the whole of the chlorine has been removed from solution, as chloride of silver, the precipitate *retains* a faint yellow tinge. The first drop of the silver solution produces a yellow precipitate, because it is for the moment in excess at that part of the liquid; but this colour disappears rapidly and entirely on agitation, so long as a trace of chlorine remains in solution.

Bromine, Br. 80.

A clear, heavy liquid of a dark red colour; density 2·97. It should be perfectly volatilizable by heat, and soluble in 34 parts of water. Impurities and adulterations are not probable.

The distinctive character of bromides is the evolution of a reddish-brown coloured vapour, when heated with binoxide of manganese, which dissolves with scarcely any colour in ammonia. Soluble bromides yield with nitrate of silver a white precipitate (AgBr), insoluble in nitric acid or ammonia.

Bromine is estimated as bromide of silver, by precipitation with nitrate of silver, in like manner as chlorine.

Iodine, I. 127.

Should be in the state of blackish scaly crystals perfectly soluble in alcohol, and should not leave any residue when sublimed. The probable adulterations are,—

Graphite, sulphuret of antimony, charcoal, sand, &c., indicated by a residue insoluble in alcohol. This may be collected by filtration, estimated, and further examined.

Water cannot be estimated in the usual manner, on account of the volatility of iodine. It may, however, be estimated by combining the iodine with an excess of mercury, and weighing the mixture, dried at 212° F. The amount of water is then indicated by the loss of weight.

The iodine to be examined should be weighed in a small capsule containing about eight times as much mercury, together with a light agate pestle. The substances are then thoroughly well mixed by rubbing together with the pestle; the capsule with its contents placed in the drying bath, and when quite dry, weighed again.

The distinctive character of iodine is the production of an intense blue colour when brought in a free state in contact with solution of starch.

This test may be applied to iodides by mixing the neutral or slightly acid solution, with starch solution, and then adding chlorine water. Great care must be taken in testing for small quantities of iodine, not to add more chlorine than will be sufficient for the liberation of iodine, since the iodine would then be converted into iodic acid, which has no action upon starch.

Iodine is estimated either as iodide of silver (AgI) precisely in the same manner as chlorine, or when it is accompanied by chlorine or bromine as iodide of palladium (PdI), by means of a solution of chloride of palladium, the brownish black precipitate being dried in a vacuum over sulphuric acid.

Iodine may be estimated volumetrically by means of the reaction between iodides and perchloride of iron,—

$$IH + Fe_2Cl_3 = HCl + 2FeCl + I.$$

The quantity of iron f, reduced to the state of protochloride by a given quantity q of the material in which iodine or an iodide is to be estimated, is estimated by means of a solution of permanganate of potash (100 cubic centimeters of which are equivalent to 0·56 Fe) according to the method described at p. 124, and the amount of iodine x calculated by the proportion—

$$x : f = I : Fe_2.$$

The quantity of material to be taken in order to obtain per-centage results, is for—

	grm.
Iodine.....	1·27
Iodide of potassium	1·66.

The experiment is made by boiling the mixture of the material in which iodine is to be estimated, with an excess of perchloride of iron, in a narrow-necked flask, so that the iodine may be volatilized, then diluting largely with water, and estimating the amount of iron f in the state of protochloride.

It is necessary that the iodine should be in combination as an iodide.

Compounds of iodine with oxidizing substances should be evaporated to dryness with excess of caustic potash, and converted into iodide of potassium by ignition.

When it is desirable to estimate the iodine directly, this

is effected by means of the reaction between free iodine and hyposulphite of soda,—

$$I + 2NaO,S_2O_2 = INa + NaO,S_4O_5.$$

The normal solution of hyposulphite is made by dissolving 24·84 grms. in water to the volume of a litre; 100 cubic centimeters of it are equivalent to 1·27 grm. of iodine, and one cubic centimeter is equivalent to 0·0127 grm.

The value of this solution varies gradually, and must be estimated from time to time with a solution of iodine in alcohol.

Experiment. In order to collect the iodine liberated by the reaction with perchloride of iron, the material must be added to the perchloride of iron in a small tubulated retort, the neck of which is drawn out, and immersed in weak solution of potash contained in a narrow-necked flask. The iodine is thus obtained in solution, $\frac{5}{6}$ths being in the state of iodide and $\frac{1}{6}$th in the state of iodate. By the addition of hydrochloric acid the whole of the iodine is again liberated, and in a fit state for estimation by means of hyposulphite of soda. A little very dilute solution of starch is then mixed with the liquid, and the normal solution added until the blue colour suddenly disappears.

When the quantity of starch added is very small, the liquid acquires a greenish brown colour, and is rendered turbid by suspended iodine. Then on the addition of the normal solution of hyposulphite it gradually becomes clear, and finally pure blue. At this point only a few drops of reagent are needed to complete the reaction, and this may be done with a decimal solution as in the estimation of silver, if it is desirable.

This method of estimation is especially adapted for the valuation of varec and kelp, the mother-liquors of salt-works, and commercial iodide of potassium.

For the valuation of iodine, and when protoxide of iron or other reducing substances are associated with the iodides to be estimated, the second process only can be adopted, but in other cases both experiments should be made together and the mean results taken.

The iodides of heavy metals must be converted into iodide of potassium by boiling with caustic potash, and their amount calculated from the iodine found.

Another method of estimating iodine is based upon the liberation of iodine from iodides by chlorine,—

$$IM + Cl = ClM + I,$$

and its subsequent conversion into iodic acid by a larger quantity of the same reagent,—

$$I + Cl_5 + 5HO = 5ClH + IO_5.$$

The reagent used is hypochlorite of soda, and as neither alkaline iodides nor iodates produce a blue colour with starch, this substance serves to indicate the end of the reaction. Five times as much normal solution will be necessary to produce perfect decoloration as may be required to produce the highest degree of coloration in the liquid under examination.

The normal solution of hypochlorite of soda is made of such value that 100 cubic centimeters are equivalent to 1·000 grm. of iodine, and the amount of iodine x is found by the proportion,—

$$x : Cl = I : Cl_6.$$

The quantity of material to be taken in order to obtain per-centage results, is for,—

	grm.
Iodide of potassium	1·299
Iodine	1·000.

Iodine cannot be weighed in exactly the proper quantity on account of its volatility, so that an approximative quantity must be taken, and the weight estimated in a light stoppered tube.

The experiment is made in the usual manner, by adding the normal solution from a volumeter to the solution of a suitable quantity of the substance in which iodine is to be estimated, rendered acid by a few drops of hydrochloric acid, and mixed with some dilute solution of starch, until the whole of the iodine is converted into iodic acid, the end of this reaction being indicated by the total disappearance of the blue colour at first produced by the liberated iodine and the starch.

For the valuation of iodine, to which this process is particularly adapted, it is dissolved in caustic potash and mixed with an excess of hydrochloric acid previous to the addition of the normal solution. The iodine in this case being un-

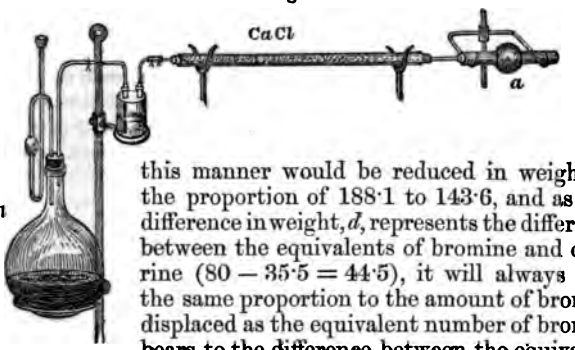
combined, only five equivalents of chlorine are requisite for its conversion into iodic acid, so that the proportion by which its amount is found, becomes—

$$x : Cl = I : Cl_5.$$

Quantitative separation of chlorine, bromine, and iodine.

The estimation of bromine, when associated with chlorine, is effected indirectly, by converting the bromide of silver in a known quantity of a mixture of bromide and chloride of silver into chloride, and calculating the amount of bromine from the difference of weight, resulting from the substitution of chlorine for bromine. The apparatus for this experiment is represented by fig. 47. Pure bromide of silver treated in

Fig. 47.



this manner would be reduced in weight in the proportion of 188·1 to 143·6, and as this difference in weight, d, represents the difference between the equivalents of bromine and chlorine ($80 - 35{\cdot}5 = 44{\cdot}5$), it will always bear the same proportion to the amount of bromine displaced as the equivalent number of bromine bears to the difference between the equivalent numbers of those substances. Thus, if in an experiment 1·0 grm. of a mixture of bromide and chloride of silver weighs after treatment with chlorine 0·85 grm., the amount of bromine x will be 0·269,

$$x : d\ (= 1{\cdot}0 - 0{\cdot}85) = 80 : 44{\cdot}5.$$

Both substances are precipitated by nitrate of silver, and the total weight of chloride and bromide of silver estimated in the usual manner. A portion of the melted mixture is then introduced into a bulbed tube *a*, its weight accurately determined, and exposed, at a temperature sufficient to render it liquid, to the action of dry chlorine gas for about twenty minutes. The tube is weighed again when cool, and the

amount of bromine calculated, as already described, from the difference between the two weights.

Iodine, associated with chlorine, may be estimated in a manner precisely similar, and the results obtained are more accurate than in the estimation of bromine, because the equivalent number of iodine differs from that of chlorine more than the equivalent number of bromine does. It may likewise be separated by precipitation, as iodide of palladium, by a solution of nitrate of palladium.

Iodine is separated from bromine also as iodide of palladium, but the reagent used for this purpose must be chloride of palladium, since there would be a partial precipitation of bromide of palladium in the absence of alkaline chlorides.

In the case most likely to present itself, iodine is associated with both chlorine and bromine. When the proportion of chlorine is small, it cannot be estimated without much trouble, but when it preponderates over the other two substances, the iodine may be separated by nitrate of palladium, in the filtrate from which palladium has been removed by sulphuretted hydrogen; the chlorine and bromine precipitated together by nitrate of silver, and their relative amounts ascertained by the indirect process.

Cyanogen, C_2N. Cy. 26.

The distinctive characters of cyanogen compounds (*cyanides*) are,—1. The evolution of hydrocyanic acid when treated with dilute sulphuric acid. 2. The formation of a dark blue precipitate (*ferrocyanide of iron*) when the slightly *alkaline* solution is mixed with a solution containing protosulphate and perchloride of iron, and then with hydrochloric acid. This test may be applied to the distillate obtained by treatment with sulphuric acid. 3. The smell of hydrocyanic acid is sufficiently peculiar to indicate the presence of very small quantities.

The cyanides, which are used for many purposes in the arts and in medicine, are mostly liable to vary in value. The method which is perhaps most suitable for the valuation of commercial cyanide of potassium, is based upon the reaction between that salt and iodine:—

$$KCy + 2I = KI + CyI.$$

The test solution is made by dissolving 25·40 grms. of

iodine in alcohol (0·864 spec. gr.) to the volume of one litre. As iodine always contains some little impurity, the value of the solution must be confirmed by means of hyposulphite of soda: 100 parts of this salt dissolved in water, absorb 51 parts of iodine, forming a colourless liquid. It is therefore easy to ascertain the actual amount of iodine in the test solution by adding it to a known quantity of hyposulphite in solution so long as its colour is destroyed. A cubic centimeter of this solution is equivalent to ·0065 grm. of cyanide of potassium, and the amount of this salt x is found in any experiment by multiplying this number by the number of cubic centimeters of test solution used:—

$$x = 0{\cdot}0065 \times C_n.$$

The quantity of material to be operated upon in order to obtain direct per-centage results is 0·652 grm.

The experiment is made by adding, from a volumeter, the test solution to the cyanide dissolved in water saturated with carbonic acid, which is requisite for the purpose of converting any caustic alkali that may be present into bicarbonate, which has no influence upon the result, while caustic alkali would render it too high by retarding the coloration of the liquid beyond the point at which the whole of the cyanide had been decomposed.

When alkaline sulphides are present, a little acetate of lead must be added to the solution of cyanide, and the sulphide of lead separated by filtration before the addition of the test solution, otherwise the liquid will be rendered turbid in the experiment by the separation of sulphur.

The method already described for the estimation of ferrocyanide of potassium may be applied to the valuation of cyanide of potassium. For this purpose it must first be converted into ferrocyanide by boiling the solution with protosulphate of iron free from persulphate or copper. The experiment is then made in the usual manner, and the amount of cyanide x calculated from the quantity of ferrocyanide y found, by the proportion—

$$x : y = KCy : K_2Cfy.$$

Another method of estimating cyanogen, or cyanides, which is especially applicable to the valuation of medicinal

preparations, is based upon the characters of a double cyanide of silver and potassium ($AgCy + KCy$) which is soluble, and is not decomposed by caustic alkali, but gives with nitrate of silver a precipitate of cyanide of silver, so that when nitrate of silver is added to a solution containing a soluble cyanide with excess of caustic potash, there is no permanent precipitation of cyanide of silver until the nitrate of silver added exceeds the proportion requisite for the formation of this soluble double cyanide, and then the cyanide of potassium yields up its cyanogen and a permanent precipitate is formed.

According to the formula of this double cyanide,

$$AgCy + KCy,$$

1·08 grm. of silver corresponds to ·52 grm. of cyanogen, and in an experiment the amount of cyanogen x is calculated from the quantity of silver s required by the proportion—

$$x : s = Cy_2 : Ag.$$

The normal solution is made by dissolving 5 grms. of pure silver in nitric acid, evaporating to dryness, and adding water to the volume of one litre. One cubic centimeter of this solution, containing 0·005 grm. of silver, is equivalent to 0·0025 grm. of anhydrous hydrocyanic acid.

The experiment is very simple: the weighed or measured quantity of hydrocyanic acid, mixed with a slight excess of caustic potash, or, the solution of alkaline cyanide, is introduced into a flask, the normal solution of nitrate of silver added until a very slight permanent precipitate appears, and the result calculated from the volume of solution required.

The great excellence of this method consists in the fact, that the presence of chlorides or formic acid—which are likely to be associated with hydrocyanic acid and cyanides—does not in the least affect the accuracy of the results, and there is moreover an advantage in the circumstance that the end of the reaction is indicated by the commencement, and not by the cessation of the precipitation, and is therefore much more easily recognizable.

In testing medicinal preparations which, like bitter almond water, are turbid, owing to the presence of suspended oil, enough water must be added beforehand to render the liquid clear.

Carbon, C. 6.

The method of estimating carbon in organic substances, although strictly speaking beyond the range of technical analysis, is applicable to a variety of cases of technical examination, and must therefore be described here. The carbonaceous substances, such as coal and other kinds of fuel, animal charcoal, &c., mostly require special methods of valuation, determined by the purposes to which they are applied.

Elementary Analysis of Organic Substances.

Carbon and hydrogen are constant constituents of organic substances, oxygen and nitrogen are frequently associated with them, and sometimes also sulphur, phosphorus, &c., in small proportion.

When it is uncertain whether an organic substance to be analysed contains nitrogen or sulphur, qualitative tests must be applied beforehand to ascertain this point, because their presence necessitates some modifications in the analytical operation. Oxygen need not be tested for, since the analysis is conducted in the same manner whether it is present or not.

Nitrogen is tested for by heating the substance in a long test-tube with powdered hydrate of potash or soda lime (p. 197). The nitrogen is thus converted into ammonia, and is indicated by the smell and the production of a blue colour in litmus paper held over the mouth of the tube.

Sulphur is tested for by projecting an intimate mixture of the substance with soda and nitrate of potash into a crucible containing nitrate of potash in a state of fusion. The presence of sulphur is indicated when the cooled mass, dissolved in water and mixed with excess of hydrochloric acid, gives a precipitate (BaO,SO_3) with chloride of barium.

Estimation of carbon and hydrogen. In all cases these elements are estimated by exposing the substance to a high temperature with a sufficient supply of oxygen to ensure complete oxidation, the products being carbonic acid (CO_2) and water (HO). These are collected apart, and from their amounts c and h, the quantity of carbon $= \kappa$ and of hydrogen $= \nu$ are calculated by the proportions—

$$\kappa : c = C : CO_2, \text{ and } \nu : h = H : HO;$$

then the quantity of substance burnt being known, the percentage of these elements may be easily ascertained.

The reagents used in this analysis are,—

1. *Oxide of copper* (CuO), as a source of oxygen, which it readily yields to organic substances at a bright red heat. It should be in a granular state, free from powder, and the fragments all about the size of small shot.

2. *Chromate of lead* (PbO,CrO_3), likewise as a source of oxygen for the combustion. It is in many cases preferable to oxide of copper.

3. *Chloride of calcium* (CaCl + 2 aq.), for collecting the water, formed by the combustion, and separating it from the carbonic acid. It should be prepared by evaporating a solution of the salt to dryness, with constant stirring, so as to obtain the salt in small porous lumps.

4. *Hydrate of potash* (KO + aq.), in solution (1·27 sp. gr.), for collecting the carbonic acid formed by the combustion; also in a solid state.

The apparatus required consists of several parts: viz.—

1. *The combustion tube, a p*, (fig. 48) of stout potash glass,

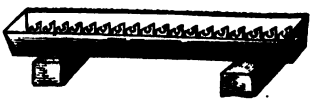
Fig. 48.

capable of bearing a very high temperature without melting. It should be about 28 inches in length, and half an inch internal diameter, with the edges made smooth by the blowpipe flame and very slightly turned outwards, so as to give additional strength that will admit of corks being fitted tightly into the ends of the tube.

This tube is two-thirds filled with oxide of copper, which is secured in its place by loose plugs of asbestos, *cc'*, at each end.

2. *The furnace* best adapted for organic analysis is represented by fig. 50, and the source of heat is ordinary coal-gas, but when this is not to be obtained, the charcoal furnace (fig. 49) must be used. It is constructed of stout sheet-iron, perforated with holes at the bottom and fitted along the middle

Fig. 49.



with a row of supports, upon which the combustion tube rests, while its anterior end projects through the hole *a*.

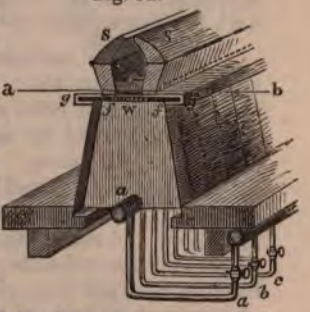
The gas furnace (fig. 50) consists of two separate pieces,

Fig. 50.

A and B, made of stout sheet-iron lined with clay.

The part below the line, **a, b,** is in A, 20 inches long, 6 inches high, 4 inches wide, and is furnished with transverse diaphragms of sheet-iron, extending from the bottom to the top, at distances of one inch apart. It is made, as shown in the section (fig. 51), to fit into a hole in the slate slab of a table upon which the whole apparatus is arranged; it is open at the bottom, and the top is covered with wire gauze, **w,** tightly fitted at each side. The part above the line **a, b,** is but one inch and a half high and two inches wide; it is furnished with transverse pieces of sheet-iron for supporting the combustion tube, and with lateral screens, **s, s,** moving upon hinges at the outer edge. It is connected with the lower part of the furnace by means of the flanges *ff*, which slide into the grooves *gg*.

Fig. 51.



The gas is supplied at *s*, through a tube with lateral branches extending upwards into the chambers formed by the transverse diaphragms and terminating in iron tubes, *a*, *b*, ... *l*, *m*, half an inch wide, closed at both ends, and perforated with two rows of holes about a quarter of an inch apart at the upper surface. The gas issuing from these tubes mixes with air in its passage upwards to the wire

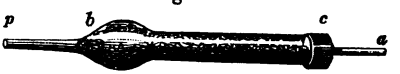
gauze, and when ignited above it, burns with a blue flame, affording a very high temperature.

It is sometimes desirable, as in the estimation of nitrogen (see p. 197), that the tube should be heated gradually, commencing at one end, and this may be effected with the gas furnace by having a separate stop-cock to each of the tubes *a*...*l*, *m*, or, by fitting into the gas tube, *s t*, an oiled leather piston which serves as a stop-cock, and when pushed into the end, prevents the passage of gas to any of the burners except *a*, but when drawn backwards opens successively the branches which supply *bcd*, &c. When the whole length of the tube is to be heated at once, as in the estimation of carbon and hydrogen, the piston is not required, and may be removed from the tube, the end, *t*, of which is then to be closed by a brass cap.

The other piece, B, of the furnace is constructed in a similar manner, but is only 6 inches long; the part below the wire gauze is divided by transverse diaphragms into four separate chambers, each fitting over one of the burners, *n*, *o*, *p*, *q*, which are furnished with stop-cocks, and supplied with gas through the tube *s*, *t*.

3. *Chloride of calcium tube* (fig. 52), in which the water is separated from the carbonic acid and collected. It should be made of light glass,

Fig. 52.



about 4 inches long and half an inch wide. A loose plug of cotton wool is inserted at *b*, so as to extend about half an inch along the narrow tube towards *p*; the bulb and straight portion of the tube are filled with fragments of chloride of calcium, another loose plug of cotton wool laid on the top, and the mouth, *c*, closed by a small cork, through which passes a small tube *a*. The cork and edge of the tube should be coated with lac varnish, to render the joint perfectly tight.

Fig. 53.

4. *Potash apparatus* (fig. 53), in which carbonic acid is

collected. It is essentially a modified form of Woulff's bottles, and consists of five bulbs, made of thin blown glass, and connected together, so that gas entering at *p* has to pass successively through the bulbs 2, 3, and 4, which are partly filled with solution of potash. The bulb 1 prevents the liquid from being forced back through *p* into other portions of the apparatus, in consequence of the rapid absorption of the gas, and 5 prevents it from being projected out through *a* when a current of non-absorbable gas is passed through the apparatus.

Fig. 54.

5. *The dry potash tube.* In some instances it is necessary to provide against an error that would affect the estimation of carbon, owing to the volatilization of water from the potash solution, when a current of hot gas is passed through the potash bulbs. This is done by making the gas issuing from *a*, pass through a tube (fig. 54) filled with fragments of hydrate of potash.

The ends, *p* and *a*, of this tube, and those of the chloride of calcium tube and potash apparatus, should be closed with very small corks when they are not in use, to prevent absorption of water or carbonic acid, from the atmosphere.

6. *Silk cord, caoutchouc tube,* and sound well-dried corks should be kept ready for making connexions.

The quantity of material to be taken for an experiment is from 0·5 to 1·0 grm. It should be, in fine powder, perfectly dried by exposure over a surface of sulphuric acid or in an air-bath at a temperature of 212° F., or upwards, until the weight remains constant.

Fig. 55.

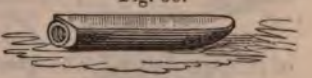
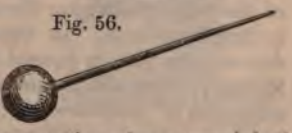


Fig. 56.



Solid substances are weighed in small trays (fig. 55) of platinum or porcelain.

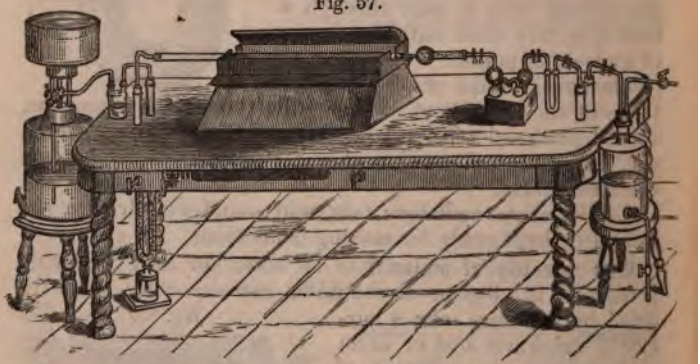
Liquids, after being dried by distillation over chloride of calcium or otherwise, are weighed in small bulbs with a long capillary neck (fig. 56). They

are filled by heating the bulb and immersing the end of the neck in the liquid, which rises into the bulb as the air contracts by cooling. When the bulb is filled, the extreme end of the capillary neck is sealed in the blowpipe flame, and the bulb weighed.

In weighing either the tray containing solid substance or a bulb with liquid, it should be enclosed in a light dry test tube, closed by a cork, both of which are of known weight, and are kept in the dry air chamber (fig. 13) for the purpose. When the exact weight of substance has been determined, the tray is placed in the dry air chamber until the combustion tube and other parts of the apparatus are ready for use.

Experiment. The combustion tube, charged with oxide of copper, is placed in the division A of the furnace with the end *a* projecting about an inch beyond the outside, as is shown in fig. 57, which represents the general arrangement

Fig. 57.



of the apparatus. The gas is lighted in A, and the portion of the tube containing the oxide of copper gradually heated to bright redness. In this state a current of dry air, or of dry oxygen gas, is passed through the tube by connecting the end *p* with the air or oxygen reservoir. By this means all traces of moisture are removed from the tube, and any reduced copper reconverted into oxide. Both ends of the tube are then closed with dry corks, and while it is cooling,

the chloride of calcium tube, potash bulbs, and potash tube are weighed separately.

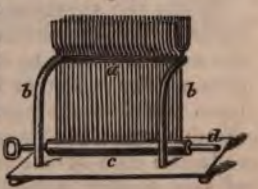
By means of a sound cork bored to fit upon the end, *p*, of the chloride of calcium tube, and into the end, *a*, of the combustion tube, these two parts of the apparatus are connected. The potash bulbs are connected with the chloride of calcium tube by a short piece of caoutchouc tube, tied with ligature silk, and the potash tube is connected with the bulbs in the same manner.

These connexions are made as rapidly as possible; when they are complete, the cork is removed from the end *p* of the combustion tube, the tray containing the substance to be analysed, slipped in with a loose plug of asbestos behind it, and a connexion made with the desiccating tube.

Before commencing the operation, it is necessary to ascertain whether all the joints are perfectly tight. This is done by removing a few bubbles of air from the apparatus by heating the bulb 1, while 4 is raised for a moment somewhat above the level of the others; if, upon cooling, the solution of potash rises into the bulb 1 and maintains its level, the connexions may be considered tight.

The portion of the tube containing the oxide of copper is then heated to bright redness; the aspirator with the cock *r* open, is attached to the end *a* of the potash tube, and after setting up a very feeble current of air through the apparatus by slightly opening the cock of the air reservoir, and heating the tube at *c*, heat is applied to the substance in the tray by means of the moveable burner. This part of the operation requires the greatest caution, and its progress must be watched without intermission, otherwise the vaporous products are given off so abundantly, that, instead of being perfectly carried along by the slow current of air through the red-hot oxide of copper, they pass backwards, and condense in the drying tubes of the gas reservoir. When this happens the experiment is spoiled, and it is therefore better to exercise a due amount of patience in the first application of heat to the substance.

Fig. 58.



The gas-burner C (fig. 58), used for this purpose, consists

of a double row of very small brass tubes, soldered to a rod *a*, and supported upon a slight stand *b*, *b*. The lower end of each pair of the tubes is connected by vulcanized caoutchouc with a similar-sized tube screwed into the side of the brass cylinder *c*, made to serve the purpose of a stop-cock by means of a moveable leather piston, which as it is drawn back from the position shown in the drawing, admits the passage of the gas supplied at *d* to any number of the tubes.

By means of this arrangement, and the cock regulating the gas supply, the decomposition of the substance is perfectly under control. The apparatus is likewise very useful for other purposes.

The vaporous products evolved are oxidized in traversing the oxide of copper, and issue from the combustion tube at *a* in the form of carbonic acid and water, which are separated and collected in the chloride of calcium tube and potash bulbs.

When the substance in the tray is perfectly charred, the other part, B, of the furnace is put in its place, the gas lighted, the cock of the oxygen reservoir is slightly opened, and the temperature of that portion of the tube raised sufficiently high to ensure the perfect combustion of the carbonaceous residue; then the current of oxygen is stopped, the reservoir disconnected, the gas turned off, and while the combustion tube is cooling, any remaining traces of carbonic acid swept out by passing a current of air through the apparatus by opening the cock *r'* of the aspirator and closing *r*. When about a litre of air has been passed through the apparatus, the potash tube and bulbs, and the chloride of calcium tube are detached and weighed as quickly as possible. The increase of weight in the former two are added together and calculated as carbonic acid, and the increase in the chloride of calcium tube calculated as water.

When the substance analysed contains inorganic constituents, they will remain in the form of ash in the tray, and the amount should be estimated as a check upon the results obtained for carbon and hydrogen; when the substance analysed is free from oxygen, nitrogen, &c., the sum of the three should be equal to the original weight of the substance.

In the case of substances containing nitrogen, a portion of this element would, on ignition with oxide of copper or chromate of lead, be evolved in a free state, while another

portion would be more or less oxidized, and the products absorbed by the solution of potash. In order to prevent the inaccuracy that would thus attach to the estimation of the carbon in the case of nitrogenous substances, the tube used for the combustion should be about 6 inches longer than usual, and some 5 inches of this space in front of the oxide of copper is filled with copper turnings, which during the experiment are kept at a *bright* red heat. In contact with copper at this temperature, the oxides of nitrogen are perfectly decomposed, and the nitrogen evolved in a free state passes out of the apparatus without influencing the estimation of the other constituents. When the substance contains sulphur, sulphurous acid is generated in the combustion; and as this would affect the accuracy of the carbon estimation in the same manner as the oxides of nitrogen, it must be prevented from entering the potash bulbs by inserting, between them and the chloride of calcium tube, another tube, of the same shape, filled with small lumps of peroxide of lead (PbO_2), which absorbs the sulphurous acid.

Estimation of oxygen. No method of estimating directly the oxygen in organic substances is known; its amount must therefore be ascertained by deducting from the original weight of the substance the sum of its other constituents.

Estimation of nitrogen. The estimation of nitrogen in organic substances requires a special experiment, and the reaction upon which it is based is the same as that by which its presence in organic substances is detected, viz. its perfect conversion, by ignition with a considerable excess of hydrate of soda, into ammonia which is collected by absorption in hydrochloric acid, and weighed in the form of chloride of platinum and ammonium ($PtCl_2,NH_4Cl$). The quantity x of nitrogen is calculated from the quantity of double chloride q obtained, by the proportion—

$$x : q = N : PtCl_2,NH_4Cl.$$

The reagents required for this purpose, are:—

1. *A mixture of hydrate of soda with hydrate of lime,* prepared by slaking lime with a solution of soda in such proportion that there are two parts of hydrate of lime to one of hydrate of soda. The mixture is ignited for some time in

an earthen crucible and preserved, in fine powder, in a well-stoppered glass bottle.

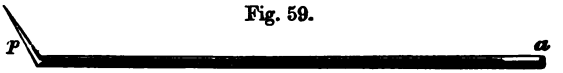
The reaction which takes place when substances containing nitrogen in any form but nitric acid are ignited with the soda-lime, consists in the decomposition of the water of the hydrate of soda, its oxygen combining with the carbon of the organic substance forming carbonic acid, while its hydrogen combines with the nitrogen, forming ammonia.

2. *Hydrochloric acid* (sp. gr. 1·13), for absorbing the ammonia produced.

The apparatus required consists of,—

1. Combustion tube, 16 or 18 inches long, sealed at the end *p*, and drawn out to a point which is turned upwards, as shown in fig. 59.

Fig. 59.



2. Bulbed tube for the hydrochloric acid (fig. 35).

Experiment. The substance is mixed in a perfectly dry mortar with sufficient soda-lime to fill one half of the combustion tube, and transferred to the tube after the end *p* has been filled about an inch high with soda-lime. The mortar is rinsed two or three times with soda-lime, enough to fill the tube within an inch of the mouth, a loose plug of asbestos placed in front of the charge, and a channel made along its entire length by knocking the tube upon a table. The bulb-tube containing hydrochloric acid is then attached by means of a sound cork fitting upon *p* and into the end *a* of the combustion-tube. Heat is applied to the tube, either by a gas-furnace, in which the supply of gas is regulated by a piston working in the gas-tube (see fig. 50, p. 192), or by ignited charcoal in the furnace (fig. 49), commencing at the anterior part, which must be kept at a bright red heat throughout the operation, and gradually extending it to the whole length of the tube, so as to keep up a moderate and continuous evolution of gas. When this ceases, and the mixture in the tube has become quite white, the hydrochloric acid rises into the bulb 1; the end *p* of the tube is

then broken with a pair of forceps, and air drawn through the apparatus by an aspirator.

The contents of the bulb-tube are poured into a porcelain basin, the adherent portion washed out with a mixture of alcohol and æther, and lastly with water; an excess of chloride of platinum solution added, the whole evaporated to dryness on a water-bath, the residue digested with a mixture of two parts of alcohol with one of æther, and the chloride of platinum and ammonium collected by filtration, washed with alcohol and æther, and weighed. When an approximative estimation of nitrogen is sufficient, instead of the hydrochloric acid, a solution of tartaric acid in *absolute* alcohol may be used for collecting the ammonia. In this case the nitrogen is estimated in the form of bitartrate of ammonia, which being insoluble in alcohol, is precipitated in the bulb-tube, and may be collected by filtration, washed with absolute alcohol, dried at 212° F., and weighed. It contains 8·4 per cent. of nitrogen.

The amount of nitrogen may likewise be estimated volumetrically by collecting the ammonia by means of the bulb-tube, in 20 cubic centimeters of the normal solution of sulphuric acid (containing 0·80 grm. SO_3), and then estimating by means of the equivalent solution of saccharate of lime (p. 74), or of carbonate of soda (p. 70), how much acid, S, remains in a free state. The quantity of ammonia, y, will be equivalent to the acid neutralized, s—

$$y : s\ (=0{\cdot}80-S) = NH_3 : SO_3,$$

and from it the amount of nitrogen x may be calculated by the proportion—

$$x : y = N : NH_3.$$

The presence of sulphur does not affect the estimation of nitrogen.

Estimation of sulphur. The sulphur in organic substances is always estimated in the form of sulphate of baryta, BaO,SO_3. The amount of sulphur x being calculated from that of the sulphate of baryta y obtained, by the proportion—

$$x : y = S : BaO,SO_3.$$

The reagent used for the oxidation is,—

Nitrate of potash mixed with two parts of *carbonate of baryta.* The substance, mixed with about 25 times its weight

of the dry powder, may be transferred to a short, narrow combustion-tube sealed at one end, and gradually heated to redness, commencing at the anterior end; or, the mixture may be projected in small successive portions into a tolerably capacious platinum crucible kept at a red heat. In both cases the product of the fusion is dissolved in water, a slight excess of hydrochloric acid added, and the sulphate of baryta collected by filtration and weighed.

Examination and valuation of coal and other kinds of fuel, animal charcoal, &c.

None of the varieties of coal consist wholly of combustible carbonaceous substance. In anthracite the proportion of earthy (ash) ingredients is sometimes extremely small, but in the bituminous or caking coal ordinarily used, it is generally much larger and moreover very variable.

The analysis of coal may be effected in the manner described above, under the head of carbon estimation, but the results thus obtained, although by no means without value, are not alone sufficient for the desired result. The general value of coal or any other kind of fuel, is dependent upon the amount of heat generated by the combustion of a given quantity; the special value for certain purposes is determined in a great measure by the absence of substances occurring in some kinds, and by the mode in which heat is generated by the combustion, and as this varies greatly for different kinds of fuel, it is always necessary, in ascertaining the value of fuel for practical purposes, to apply some more direct test of its capabilities than the elementary analysis.

The physical characters are to a considerable extent indicative of the applicability of fuel to certain purposes, and these, together with the actual heating effect of a given weight, as well as the per-centage of ash and water, furnish in many instances the best data for estimating its value.

Estimation of water. Coal and similar kinds of fuel should be very finely powdered, and a weighed quantity exposed to a temperature of 212° F. until no further decrease of weight takes place. It should then be exposed to a higher temperature, because very frequently the water is not otherwise separable. The dried coal must be allowed to cool in the

dry air chamber and weighed in a covered vessel, and the amount of loss calculated for 100 parts of the fuel.

Wood, turf, &c., are dried in the form of chips at a temperature of 212° F.

Estimation of ash. In the case of coal, a weighed quantity of the powdered substance is ignited in a wide porcelain crucible, the heat being gently applied in the first instance, so that none of the powder may be forced out by disengaged gas. When the carbonization is complete, the crucible is laid somewhat on one side, so as to facilitate the access of air, and a strong heat applied to the crucible by means of the gas furnace (fig. 16) until all traces of carbon are perfectly burnt away. The residue is then weighed, and the percentage of ash calculated. It is always advisable to make two or more experiments, and to take the mean result.

Estimation of the calorific power of fuel. There is no means of estimating the actual amount of heat generated by combustion, and therefore the capability of fuel in this respect can only be ascertained relatively. It is generally expressed by the number of pounds of water that may be heated from 32° to 212° F. by the combustion of a given quantity, by weight or volume, of the fuel. The effect produced by the combustion of pure carbon is taken as the standard of comparison, and that amount of heat which raises the temperature of a pound of water one degree, as the unit of heat. This estimation is attended with considerable difficulties, in the exclusion of all disturbing influences which might render the results incomparable with each other.

The experiment may be conducted in a small furnace, the chimney of which is connected with a long spiral tube, immersed in a large vessel filled with water at a known temperature; but perhaps the best mode of making such experiments, when they are at all applicable, is to burn a given quantity of the fuel in a furnace of suitable construction, and observe how much water can be evaporated by it. The fuel should be added only in small quantities, sufficient to keep up a steady evaporation without waste of heat. The experiment should be continued for six or eight hours, and the boiler kept filled meanwhile, to a constant level by a very gradual supply from a known quantity of water at a known

temperature. The result thus obtained is always under the true value in consequence of loss of heat by radiation, but if the experiment is well managed this loss will be tolerably constant.

From the quantity of water evaporated by the combustion of a given quantity of the fuel, an approximative indication of its calorific power, as compared with other kinds, may be obtained. The experimental results thus obtained may easily be reduced to a common form of expression, for the quantity of heat requisite for converting one pound of water at 212° F. into steam under the ordinary atmospheric pressure is equal to that required for raising the temperature of 5·5 pounds of water from 32° to 212° F.; so that if, in an experiment to ascertain the calorific power of turf, it has been found that for every pound of fuel burnt, four pounds of water have been evaporated, the quantity of water x that would be heated from 32° to 212° F. by the combustion of the same quantity is found by a simple multiplication,—

$$x = 5{\cdot}5 \times 4 = 22 \text{ pounds.}$$

The result may also be expressed in heat-units; for as equal quantities of heat are required to raise x pounds of water y degrees, or y pounds of water x degrees, the number of pounds of water heated from 32° to 212° by the combustion of one pound of fuel, multiplied by 180, will give, x', the number of pounds of water in which the temperature would be raised one degree, or the number of heat-units generated; thus in the above instance,—

$$x' = 22 \times 180 = 3960.$$

The quantity of water of which the temperature may be raised any given number of degrees between 32° and 212° is found in the same manner; thus the quantity of water heated by the combustion of a pound of fuel will be inversely proportionate to the number of degrees that the temperature is raised, because the quantity of heat capable of heating 22 pounds of water from 32° to 212° F. (180 degrees) will raise the temperature of 25·88 pounds from 59° to 212° F.

$$22 \times 180 = 25{\cdot}88 \times 153.$$

The following table gives the calorific power of several combustible substances used as fuel.

	Quantities of water heated by the combustion of one pound of fuel, from 32° to 212° F.	from $t°$ to $t°+1°$ F.	Relative calorific power for equal weights.	for equal volumes.
Hydrogen	236	42,480	3·03	·0077
Oil	90	16,200	1·15	30·2000
Æther	80	14,400	1·02	21·1000
CARBON	78	13,640	1·00	100·0000
Coal-gas	76	13,680	0·70	
Charcoal	75	13,500	0·96	4·9400
Alcohol	67	12,060	0·86	19·8000
Coal	60	10,800	0·77	33·0000
Wood (dry)	36	6,480	0·46	5·2600
„ (with 20 per cent. moisture)	27	4,860	35	4·9100
Turf	27	4,860	35	

The calorific power for equal volumes is found by multiplying the number expressing the calorific power for equal weights by the specific gravity of the substance.

It may for practical purposes be assumed that the calorific power of hydrogen is three times as great as that of carbon; consequently the calorific power of fuel will be proportionately greater, the greater the amount of hydrogen it contains, so that when the per-centage composition of fuel is known, its calorific power may be ascertained by calculation from these data.

For fuel consisting solely of carbon and hydrogen, the calorific power x is found by the following formula:—

$$x = 3H + C,$$

in which H represents the per-centage of hydrogen, C that of carbon.

In the case of fuel containing besides carbon and hydrogen, a certain amount of oxygen which must be regarded as if already combined with equivalent quantities of carbon or hydrogen, as carbonic acid or water, a corresponding allowance must be made in the calculation, and as oxygen is combined in carbonic acid with $\frac{3}{8}$ of its weight of carbon, and in water with $\frac{1}{8}$ of its weight of hydrogen, the formula for the calorific power will be either:—

$$x = 3H + C - \frac{3}{8}O,$$

or,
$$x = 3\left(H - \frac{1}{8}O\right) + C.$$

in which O represents the per-centage of oxygen.

The calorific power of fuel may likewise be ascertained from the amount of oxygen required for its perfect combustion. Thus carbon requires for combustion to carbonic acid, two equivalents of oxygen, hydrogen for combustion to water, one equivalent; and as the equivalent numbers of these elements are as 6 to 1, the quantities of oxygen required for perfect combustion of equal weights will be for:—

6 pounds of carbon.. 2 equivs. oxygen = 8 × 2 = 16,
6 pounds of hydrogen 6 equivs. oxygen = 8 × 6 = 48,

so that the amount of oxygen required for the perfect combustion of hydrogen, is three times as great as that required for the combustion of an equal weight of carbon. Thus the calorific powers of carbon and hydrogen bear the same relation to each other as the quantities of oxygen consumed in the combustion of equal quantities.

It follows from this, that for a certain quantity of oxygen consumed in combustion, there is always the same amount of heat generated. For as

Carbon requires for combustion $2\frac{2}{3}$ its weight of oxygen,
Hydrogen requires for combustion 8 its weight of oxygen,

while by the combustion of equal weights of

Carbon.. 78 parts of water are heated from 32° to 212° F.,
Hydrogen 236 parts of water are heated from 32° to 212° F.,

it follows that the quantity of water heated from 32° to 212°F. by the consumption of equal weights of oxygen will be—

in the combustion of Carbon .... $\frac{78}{2\frac{2}{3}} = 29{\cdot}25$,

in the combustion of Hydrogen.. $\frac{236}{8} = 29{\cdot}50$,

so that the calorific effect of oxygen may be expressed in round numbers as 30, and in heat-units as 5400 (=30 × 180).

The mode in which this relation between the calorific power and the amount of oxygen consumed in combustion is applied to the valuation of fuel, consists in exposing it to the joint influence of a high temperature and of some substance which does not by mere heating lose oxygen, while it readily yields it to the fuel, and at the same time admits

of the oxygen thus lost, being estimated. The substance used for this purpose is oxide of lead (PbO).

Experiment. One gramme of the finely powdered fuel is mixed with 40 grms. of oxide of lead, introduced into a large earthen crucible, and covered with 30 grms. more oxide of lead. The crucible, which should not be more than half-full, is covered with a lid and placed in a furnace, from which greater part of the fuel has been removed, and surrounded with hot fuel, leaving the upper part free, so that the progress of the operation may be observed by removing the lid. The heat is very gradually raised until the mixture begins to swell up in consequence of the disengagement of carbonic acid, and when this is at an end, the crucible is covered and kept for about ten minutes at a sufficiently high temperature to make the reduced lead melt into a single mass. The crucible is then removed from the furnace, and when cold broken, the button of lead separated from the melted oxide of lead, and weighed.

Pure carbon treated in this manner would reduce to the metallic state 34·56 times its weight of lead, and the amount of lead reduced by any fuel in the same manner is directly proportionate to its calorific power.

This process is applicable for the valuation of most kinds of fuel, but more particularly such as are highly carbonaceous, as anthracite. Bituminous substances cannot be examined in this way, because there is too great a loss by volatilization.

The results obtained are generally below the truth, but when the experiment is not well managed, oxide of lead may remain mixed with the lead, and thus render the result too high.

Animal (bone) **charcoal.**

Bone charcoal is principally employed as a decolorizer in refining sugar, &c. In the usual state it contains from 80 to 85 per cent. of inorganic substance. The probable adulterations are,—

Charcoal from ferrocyanide factories, } indicated by a small
Wood charcoal, } per-centage of ash.

Iron scales, schist, and earthy substances, &c., indicated by analysis of the ash (see Chap. IX.).

The valuation of animal charcoal must be effected by some process capable of indicating its relative decolorizing and absorbent powers. This is effected by means of a solution of caramel (burnt sugar). A quantity of the charcoal to be examined is placed in a filter, and the caramel solution passed through until it is no longer decolorized; a similar experiment is made with an equal quantity of charcoal of known good quality and the same solution of caramel. The relative value of the charcoal will be indicated by the amount of solution decolorized.

The apparatus used for estimating degree of colour in the liquids is described in Chapter XI.

The absorbent power of animal charcoal may be estimated by means of a solution of saccharate of lime of known volumetric value. For this purpose, 50 grms. of the charcoal are boiled with 100 cubic centimeters of the saccharate solution in a flask for about an hour, the liquid separated by filtration, and the volume of normal acid required for neutralization ascertained in the usual manner (p. 74). The difference between this result and that representing the original value of the saccharate solution, indicates the absorbent power of the charcoal.

When several samples of animal charcoal are treated in the same manner, under similar conditions, the respective results will show the relative values of the charcoal.

CHAPTER IX.

SPECIAL METHODS OF ANALYSIS, QUALITATIVE AND QUANTITATIVE, ADAPTED FOR THE EXAMINATION OF PARTICULAR CLASSES OF SUBSTANCES.

I. Examination of pigments.

It will frequently happen that the external characters and general history of substances will indicate that they belong to some one or other class of analogous substances whose constituents are with little exception few and constant, and in many instances the object of the analysis is rather to determine their proportion than their nature.

For such classes of substances special methods of analysis may be adopted which provide only for the detection or separation of such constituents as are likely to be present in substances of any particular class.

The most simple means of ascertaining the chemical nature of pigments, together with the reactions characteristic of the several kinds, are contained in the following table. The hydrochloric acid and caustic soda tests may be applied in ordinary test tubes to the raw materials as they occur in commerce, or when they have been used as paint, &c., by moistening the surface with these reagents. The incineration test may be made in a shallow platinum capsule or upon platinum-foil, either over a gas flame or with the aid of the blowpipe, as the occasion may require. In the examination of mixed pigments it is frequently necessary to separate the liquid vehicle—oil, varnish, alcohol—as far as possible by filtration, and if there is any reason to suppose that the portion still adhering should interfere with the indications of the tests applied, it must be removed by means of an appropriate solvent, such as a mixture of alcohol and æther.

Name, composition, and synonyms.	*Behaviour with hydrochloric acid.*	*Behaviour with caustic soda.*	*Behaviour on ignition.*	*Special characteristics.*
1. Ultramarine.				
Sulphuret of sodium, sulphuret of iron, and silicate of alumina. SYN. *Lapis lazuli.*	Colour destroyed with evolution of sulphuretted hydrogen.	No change.	The colour loses its brilliance.	Not poisonous.
2. Thénard's blue.				
Oxide of cobalt and alumina. SYN. *Cobalt ultramarine. Ultramarine blue.*	No change.	No change.	No change.	Not poisonous, except when containing an accidental admixture of arsenic.
3. Smalt.				
A glass containing oxide of cobalt. SYN. *Azure blue. Saxon blue.*	Becomes greenish-yellow when boiled.	No change.	No change.	Scratches glass, while Thénard's blue does not. Almost always poisonous.
4. Prussian blue.				
Compound of cyanogen with iron in proportions ranging accord-	Becomes green by boiling, and then dissolves with a yellow	Loses its blue colour and leaves a brown residue.	In the oxidizing flame leaves a red residue.	Chinese blue exhibits a cupreous lustre on the fresh frac-

oxide of zinc. SYN. *Paris, Berlin, mineral, and Chinese blue.*				
5. Mountain blue. *a.* Native. Azurite; hydrated basic carbonate of copper. *b.* Artificial; the same with earthy admixtures. SYN. *Mineral blue, English, Hamburg, Cassel, and Neuwied blue.*	Effervesce and dissolve to a yellowish-green liquid.	Become black, especially when heated.	Colour flame green, and leave a black residue.	The solution in hydrochloric acid gives on addition of ammonia a deep blue solution. Poisonous.
6. Indigo. Used only as a water colour.				
a. Indigo carmine, prepared by precipitating the sulphuric acid solution with potash. *b.* Starch nodules, coloured with indigo. SYN. *Washing or stone blue.* *c.* Solution of indigo in sulphuric acid. SYN. *Sulphate or tincture of indigo.*	Unaltered.	Become yellowish-brown. Blue restored again by acids.	Burn: only *a* leaves a considerable ash.	Water dissolves *a*, *b*, and *c*. Nitric acid destroys the colour with the aid of heat. Not poisonous.

Name, composition, and synonyms.	*Behaviour with hydrochloric acid.*	*Behaviour with caustic soda.*	*Behaviour on ignition.*	*Special characteristics.*
7. Lichen blue. Litmus. Cakes consisting of gypsum or chalk, impregnated with the colouring substance of several lichens.	Becomes red and dissolves partially.	Unaltered.	Burns, leaving as residue the earthy admixtures.	Partly soluble in water. The solutions are less deep coloured than that of indigo. Not poisonous.
		GREEN PIGMENTS.		
1. Verditer. Silicate of proto- and peroxide of iron, with potash and magnesia. SYN. *Stone green, Cyprian, Tyrolese, Bohemian, Veronese earth, French green.*	Gives a brownish-yellow solution on heating.	Becomes somewhat darker.	Brownish-red.	Not poisonous.
2. Green ultramarine. Composition similar to	Colour destroyed with	Unaltered.	When strongly heated	

a. **Bremen green.** (Porous bluish-green fragments.) **Hydrated oxide of copper, frequently mixed with gypsum.** *b.* Brunswick green and mountain green, similar to the Bremen green; brighter and mixed with heavy substances. *c.* Verdigris. Basic acetate of copper.	Give a yellowish-green solution and a white sediment.	Become black on boiling.	Colour the border of the flame greenish and become black; with soda upon charcoal give copper spangles.	Ammonia added in excess to the solution in hydrochloric acid produces a deep blue. Verdigris gives off acetic acid vapour when heated with hydrochloric acid. Poisonous.
4. Arsenical cupreous pigments. *a.* Swedish green. Arsenite of copper. Syn. *Scheele's green. Mineral green.* *b.* Schweinfurt green, or English green. Arsenite and acetate of copper.	Give a greenish-yellow solution.	Become bluish, on heating red, orange-red, or yellow.	Become black; melt (except when mixed with diluting substances) and give off white garlic vapours. The flame acquires a green colour.	Very poisonous.

Name, composition, and synonyms.	*Behaviour with hydrochloric acid.*	*Behaviour with caustic soda.*	*Behaviour on ignition.*	*Special characteristics.*
5. Green cinnabar.				
Prussian blue with chrome yellow. SYN. *Oil green, chrome green, Naples green.*	Becomes greenish-blue, and gives a solution which, when the hydrochloric acid is concentrated, deposits white crystals.	Becomes yellow.	Gives a brown or brown-yellow residue after incineration.	Somewhat poisonous.
6. Sap green.				
Alumina lake from Buckthorn juice. SYN. *Vert de vessie, Blasengrün.*	Becomes reddish-brown.	Brownish-green.	Gives a white ash.	Not poisonous.
7. Quercitron green.				
The yellow colouring substances precipitated with sulphate of copper and caustic soda.	Gives a brownish-green turbid solution.	Gives a yellow solution and black residue.	Burns, leaving a black ash; with soda upon charcoal gives copper spangles.	Poisonous.
8. Borate of copper.				
	Gives a clear yellowish-green solution.	Gives a clear solution and a black residue.	Melts. No change of colour.	Poisonous.

1. Umber. Clay with hydrated peroxide of iron and hydrated peroxide of manganese. SYN. *Turkish, Cyprian, and Sicilian umber.*	Dissolves almost entirely.	Becomes somewhat darker.	Becomes brownish-red.	Not poisonous.
2. Cologne umber. Earthy brown coal. SYN. *Van Dyk brown, Spanish brown.*	No change.	Dissolves partially to a brownish-black liquid.	Burns readily, leaving in most cases very little ash.	Not poisonous.
3. Wad. Impure superoxides of manganese. SYN. *Manganese brown, mineral bistre.*	Dissolves with disengagement of chlorine.	No change.	Becomes black.	Gives a violet bead when melted with borax.
4. Soot. Vegetable carbon. SYN. *Lamp-black.*	No change.	No change.	Burns without residue.	Not poisonous.
5. Bone black. Bone carbon. SYN. *Ivory black.*	No change.	Effervesces slightly.	Leaves a considerable white ash.	Not poisonous.
6. Graphite. Mineral carbon. SYN. *Plumbago, black lead.*	No change.	No change.	Little altered.	Not poisonous.

YELLOW PIGMENTS.

Name, composition, and synonyms.	*Behaviour with hydrochloric acid.*	*Behaviour with caustic soda.*	*Behaviour on ignition.*	*Special characteristics.*
1. Chrome yellow and chrome orange.				
Chromate of lead. Various shades, most of them containing admixtures of white substances. SYN. *Paris, Leipzig, Zwickau, Gotha, Hamburg, Cologne, Imperial, citron, and new yellow.*	Gives a green solution with a white sediment, which dissolves in a considerable quantity of water.	Becomes orange on boiling, dissolves with a yellow colour, and frequently leaves a white sediment.	Becomes reddish-brown. Gives beads of lead when melted with soda upon charcoal.	On account of the admixture of earthy substances, clay, heavy spar, &c., the solution in hydrochloric acid may be imperfect. See chrome compounds. Poisonous.
2. Cassel yellow.				
Oxide and chloride of lead. SYN. *Mineral, Montpellier, Paris, Veronese, chemical, and Turner's yellow.*	Becomes orange and, after a time, white.	Becomes lighter, dissolving partially to a yellow liquid.	Gives a white vapour and incrustation on charcoal, leaving soft beads of lead.	Poisonous.
3. Naples yellow.				
Antimoniate of lead. SYN. *Naples earth.*	Becomes first orange and then white.	Becomes reddish yellow.	Gives a white vapour and leaves brittle beads on charcoal.	Poisonous.

Syn. Mineral, Verdition, *Oriental, and Spanish yellow.*		down a yellow precipitate.	poisonous) having a sulphurous and garlic odour.	
5. Yellow ochre.				
Clay with hydrated oxide of iron.	Gives a yellow solution.	Becomes brownish.	Becomes red or reddish-brown.	Not poisonous.
Syn. *Terra di Sienna.*				
6. Gamboge.				
A gum resin.	No change.	Dissolves with brownish-yellow colour.	Melts and burns.	Forms an emulsion with water. Soluble in alcohol and æther. Poisonous.
7. Ultramarine yellow.				
Chromate of baryta.	Dissolves. The solution gives a white precipitate with sulphuric acid.	No change.	Little altered.	Little used; poisonous.
8. Massicot.				
Oxide of lead.	Becomes whitish.	Dissolves partially on boiling.	Gives beads of lead.	Poisonous.

RED PIGMENTS.

Name, composition, and synonyms.	*Behaviour with hydrochloric acid.*	*Behaviour with caustic soda.*	*Behaviour on ignition.*	*Special characteristics.*
1. Cinnabar. Sulphuret of mercury. SYN. *Chinese vermilion.*	No change.	Becomes yellowish on boiling.	Volatilizes and gives off sulphurous acid.	Moistened with solution of ammonio-nitrate of silver it becomes dark brown or black; poisonous.
2. Red lead. A peroxide of lead (PbO). SYN. *Minium.*	Becomes white and disengages chlorine.	No change.	Gives lead beads.	With nitric acid it becomes dark brown. Poisonous.
3. Chrome red. Basic chromate of lead.	Behaves in all respects like chrome yellow.			
4. English red. Peroxide of iron, frequently mixed with clay. SYN. *Red-bole, burnt ochre, rouge, stone red.*	Dissolves slowly with a yellow colour.	No change.	Becomes reddish-brown, rarely black-brown.	Not poisonous.
5. Carmine. The red colouring substance of cochineal [illegible].	Dissolves with a yellowish colour.	Gives a violet solution.	Burns, leaving a very small white ash.	Should dissolve perfectly in ammonia. Not poisonous.

[redacted]d vegetable and animal substances obtained by treating the decoctions with soda and alum.	Gives a turbid solution of a yellow colour.	Gives a red solution.	Burns, leaving a rather considerable white ash.	
a. from cochineal. *b.* from the colour of gum lac. *c.* Madder lake. *d.* Logwood.	Yellow.			
Syn. *Florentine, Vienna, Venetian, scarlet, and crimson lake.*				

WHITE PIGMENTS.

1. White lead.				
Carbonate and hydrated oxide of lead.	Dissolves with effervescence; the concentrated solution deposits small white crystals.	Dissolves; the inferior sorts leave a residue of clay, gypsum, chalk, sulphate of baryta, sometimes amounting to 75 per cent.	Becomes yellow and gives lead beads.	Dissolves completely in diluted nitric acid; a white residue indicates adulteration with sulphate of baryta. Poisonous.

Name, composition, and synonyms.	*Behaviour with hydrochloric acid.*	*Behaviour with caustic soda.*	*Behaviour on ignition.*	*Special characteristics.*
2. Zinc white. Oxide of zinc.	Dissolves without effervescence.	Dissolves without residue.	Becomes yellow and white again on cooling.	Gives a green colour when moistened with nitrate of cobalt and heated before the blowpipe.
3. Chalk. Carbonate of lime.	Dissolves perfectly with effervescence.	No change.	Apparently unaltered. The residue gives a brown colour to moistened turmeric.	Not poisonous.
4. Bone earth. SYN. *Prepared hartshorn.*	Dissolves only with the aid of heat and effervesces only at first.	No change.		Not poisonous.
5. Sulphate of Baryta. SYN. *Mineral white.*	No change.	No change.	Mixed with charcoal powder, gives a grayish mass which disengages sulphuretted hydrogen when moistened with acids.	Very heavy. Not poisonous.
6. Gypsum. Hydrated sulphate of lime.	No change.	No change.	Gives off water.	

II. Analysis of soils, marl, limestone, clay, plant-ashes, well, spring, and mineral water, etc.

There is a general similarity in the chemical nature of all substances of this kind; their constituents are seldom very varied or numerous, and generally belong to the following groups:—

Bases.	*Acids.*
Lime.	Silicic.
Magnesia.	Carbonic.
Alumina.	Sulphuric.
Oxides of iron.	Phosphoric.
„ „ manganese.	
Potash.	Chlorine.
Soda.	

The mode of combination may be more or less complex, and the substance to be analysed may be either chemically homogeneous, like glass and some native siliceous minerals, or, as is more frequently the case, a mixture.

The complete analysis of some of these substances, for instance a soil or mineral water, besides being a very lengthy operation, requires considerable experience and most careful manipulation. However, as it may safely be assumed that, with some few special exceptions, the constituents of all these substances belong to the above groups, a general system of treatment may be laid down as applicable to all.

A. Preliminary operations, etc.

Except in the analysis of some native minerals, furnace slags, &c., a complete and separate qualitative examination is unnecessary, because the presence or absence of the several constituents likely to be present in the substances, to which the system of analysis relates, are sufficiently indicated in the course of the quantitative analysis.

Sometimes, as in the analysis of soils and water, it is requisite to give attention to certain physical characters, as well as to the chemical composition. The analysis of some substances of this class indeed involves a number of special operations which are superfluous in the case of others, but

it will be easy, from the following general illustration of the analytical system, to perceive where this is the case.

Preparation of substances for analysis.—As a general rule, it is requisite that the substance to be analysed should be reduced to the state of fine powder. This may generally be effected in a porcelain or agate mortar. Large lumps of hard substances may be broken most conveniently when wrapped in two or three folds of stout glazed paper and struck with a heavy hammer. The small fragments may then be powdered in an agate mortar.

Soils being in all instances varying mixtures of more or less finely divided substances, originating from the disintegration of rocks, and consisting of gravel, sand, silicates, earthy carbonates, sulphates, phosphates, oxide of iron, &c., with organic substances and ammoniacal salts; due care must be taken in collecting the specimen for analysis, that it may represent correctly the piece of land to which the results of analysis are to apply. Sometimes there may be special circumstances to be considered, but generally speaking it is best to collect portions from different parts of the field and mix them together.

As it is obvious that the proportion of coarse pebbles to the pulverulent portion of the soil will have a great influence upon its general characters, the object of analysis would not be attained by powdering the whole together, but some mechanical means of separation must be adopted, and at the same time the relative amount of the several parts ascertained.

For this purpose a known quantity of the soil, amounting to several pounds, is first passed through a wire sieve with meshes one-eighth of an inch in diameter, which retain the gravel and larger fragments of decayed plants, &c.

Then by means of a sieve with meshes one-thirty-sixth of an inch in diameter, a further separation of coarse particles of sand, &c. is effected, and in this way the soil is separated into three portions, viz.—

a. Gravel, which is washed with water, weighed, and examined as to whether it consists wholly or in part of limestone, gypsum, slate, granite, quartz (sand), felspar, &c.

β. Coarse sand, &c., which is also washed, and the amount of organic substance estimated by burning (see p. 228).

γ. *True soil*, which passes through the finer sieve and is preserved for chemical analysis.

The mineral substance (ash) of plants is obtained for analysis by burning the well-wiped (not washed) portions of the plant in a clean clay crucible, or on a tile. The temperature should be kept as low and uniform as possible. The perfect separation of the carbon is sometimes very tedious; it may be facilitated towards the end, either by moistening the ash with nitric acid and again heating it gently when dry, or by mixing with it a little oxide of mercury and applying heat until all the mercury is volatilized.

The per-centage amount of ash is always to be ascertained by a separate experiment with a known quantity (50 to 100 grms.) of the dried plant.

In the examination of natural water much depends upon the character of the water and the object for which it is undertaken.

With regard to the former of these conditions, and from a practical point of view, water as it occurs naturally, may be of two kinds:—

1. The water of surface-springs, rivers, and wells, containing but a small amount of mineral or other substances.

2. The water of deep springs, of the sea, and of some lakes, so highly charged with saline and gaseous substances, as to be unfit for dietetic and technical uses.

The substances present in water are for the most part derived from the strata through which it passes, and they vary according to the nature of these strata, although more in quantity than in quality. These substances are principally calcareous, magnesian, and alkaline salts of sulphuric and carbonic acids and chlorine; gases derived from the atmosphere and organic substance.

True mineral water contains the same substances, but in larger amount, together with silica, iron, free carbonic acid, alkaline iodides, bromides, and sulphuretted hydrogen.

In collecting water of the former kind for examination, several half-gallon stoppered, glass bottles should be filled in such a manner as to avoid the introduction of any accidental impurity, each of the bottles labelled, and its stopper secured by string and sealing-wax.

A mineral water should be collected in the same manner,

and for the qualitative analysis from one to six or eight gallons should be collected according to the amount of saline substance it contains. When circumstances permit, this quantity may be at once evaporated to a small bulk, and the residue, together with the deposit, carefully transferred to a stoppered bottle for further examination in the laboratory.

The water should likewise be examined at the spring with litmus paper, and the temperature both of the water and air observed.

When the water is aërated, provision must be made at the spring for the estimation of the gases, as described at p. 224.

Determination of specific gravity.—The methods of estimating the specific gravity of substances are described in the Appendix.

In the analysis of soils the *apparent specific gravity* is frequently required in order to learn how much of some particular constituent, indicated by the chemical analysis, is contained in a certain superficies and depth of soil, and because the retentive power of the soil for water may be ascertained from it. The estimation is made by filling a vessel of known capacity for water with the well-dried soil, without pressing it down, and then weighing.

Estimation of the retentive power of soils for water is ascertained by pouring water upon about 200 grms. of the soil contained in a funnel, the neck of which is loosely stuffed with cotton wool, leaving it covered with a glass plate until no more water comes through, then weighing a portion of the moist soil, and estimating the water as below. It is easy to calculate from this how much water can be retained by a certain cubic extent of soil.

Fig. 60.

Estimation of water.—Adherent or hygroscopic water is estimated by exposing a known quantity of the substance (for soils, 20 grms. of the sifted soil γ) for some time to a temperature of 212° F. in the steam bath (fig. 60), and weighing several times until there is no longer any loss of weight. The per-centage of water is calculated from the loss.

A number of substances retain, even after exposure to the temperature of 212° F., a certain amount of water which is separated only at a much higher temperature. It is estimated from the loss by igniting the substance in a platinum or porcelain crucible.

Organic substance.—This, as well as the total amount of fixed mineral substance, is estimated by igniting in a crucible 20 or 30 grms. of the substance, dried at 212° F. The crucible should be capacious and loosely covered, and heat should be applied very gradually at first, especially if the proportion of organic substance is considerable, because some portion of the substance may be forced out of the crucible when the evolution of vapour is violent. The combustion should be regulated by keeping the crucible more or less covered with its lid, and towards the end of the operation it may be facilitated by stirring the substance with a thin platinum wire.

The loss of weight by the ignition of a soil is chiefly owing to the decomposition of organic substance, but in part it represents water separable only at a temperature much higher than 212° F. It is likewise possible that a small quantity of carbonic acid may be expelled from carbonate of lime or magnesia. Error from this source may be avoided by adding to the ignited substance a few drops of carbonate of ammonia solution, and again heating it very slowly until the bottom of the crucible begins to be red-hot.

Carbonic acid.—In the analysis of limestone, marl, and soils, &c., the total amount of carbonic acid must be estimated by a special experiment with the apparatus (fig. 34), and in the manner described at p. 66. 2 grms. of limestone, 3 of marl, and 5 or 10 grms. of a soil are the quantities to be taken.

The result serves as a control of the direct estimation of lime and magnesia.

The total amount of carbonic acid in mineral waters must be estimated with water direct from the spring.

The carbonic acid of aërated water must be estimated by special experiment. For this purpose five or six pint bottles, each containing about 2 ounces of chloride of barium solution and ammonia, are filled with a known volume of

the water, measured by means of a pipette (fig. 61), and the stoppers secured.

Fig. 61.

The precipitate produced in this way will contain the total amount of carbonic acid; the quantity of which may be estimated in the usual manner as directed above, or the precipitate is dried, ignited, and weighed. Then from this weight the weight of the ignited residue left by the evaporation of an equal volume of water is deducted, together with the sulphate of baryta equivalent to the sulphuric acid of the same volume; the remainder represents the carbonate of baryta, and from it the quantity of carbonic acid may be ascertained by the proportion,—

$$x : y = CO^2 : BaO,CO^2.$$

If necessary the mean of several experiments is taken.

When the water contains sulphuretted hydrogen, either alone, or together with carbonic acid, an additional number of bottles, each containing some solution of arsenous acid in hydrochloric acid, must be filled with the water, collected in the same manner.

The sulphuretted hydrogen is estimated by weighing the tersulphide of arsenic, dried at 212° Fahr., and calculating from the quantity y obtained, the amount of sulphuretted hydrogen x, by the proportion:—

$$x : y = HS : AsS^3.$$

Estimation of hardness and alkalinity of water.

The earthy salts which are generally present in water, to a greater or less amount, communicate to it a character called *hardness*, which is highly objectionable when exceeding a certain limit. For this reason the hardness must always be estimated, in the examination of water available for general purposes.

The method by which the hardness of water is estimated, is volumetrical, and is based upon the fact, that when pure water is mixed with an alcoholic solution of soap, even in very small quantity, and shaken for a few minutes, a froth is produced, which remains persistent for five minutes; while on the contrary, when the water contains calcareous or magnesian salts in solution, no such froth is produced, until a

larger amount of soap solution is added. Moreover, the quantity of soap solution requisite to produce the froth, is, to such an extent, proportionate to the amount of earthy salts present in the water, that, with a soap solution of known volumetrical value, the hardness of water is indicated by the volume required to produce the froth in a given quantity of the water.

The hardness of water is always expressed in degrees, representing one grain of carbonate of lime (CaO,CO^2) in the gallon (70·000 grains), so that water containing in solution $\frac{1}{70·000}$ by weight of carbonate of lime, or the equivalent quantities of any other calcareous or magnesian salt, would have one degree of hardness. Water containing sixteen times as much, or $\frac{1}{4375}$, would have sixteen degrees of hardness, and so on.

The normal solution is prepared by dissolving about 437 grains of white curd soap in a gallon of dilute alcohol (sp. gr. 0·92). Its volumetrical value is ascertained by direct experiment with pure carbonate of lime; and it should be such, that exactly 340 grain measures may suffice to produce the froth with 1000 grain measures of a solution having 16° hardness. For this purpose a standard lime-solution is prepared, by dissolving 16 grains of carbonate of lime in hydrochloric acid; the solution is evaporated to dryness, to remove excess of acid, the residue dissolved in water, to the volume of one gallon, and the solution preserved in a well-stoppered bottle, labeled "lime-standard 16° hardness." By diluting this standard solution in due proportions, other standards, varying from one degree upwards, may be prepared at pleasure.

To produce a persistent froth, even with pure water, a slight excess of soap solution is requisite, so that the experimental results must always undergo a certain correction before the hardness of the water is calculated from them.

The following table gives the quantities of soap solution, requisite to produce the froth in pure water, and in water with hardness varying from one to sixteen degrees.

Experiment. The standard solution, or the water to be examined, is measured with a pipette, gauged to deliver 1000 grain measures, and poured into a stoppered bottle of about

double that capacity. The normal soap solution is measured from a small burette (fig. 62), containing from 200 to 400 grain measures of water, and graduated into "test measures" of 10 grain measures each. It is added gradually, and the liquid well shaken after each addition, until a persistent froth appears upon the surface. A second experiment is then made comparatively, with the water and the standard of hardness to which it approximates most closely.

Fig. 62.

Supposing that the 1000 grain measures of water examined, required, for the production of the froth, 18 test measures of soap solution, or any one of the quantities in the second column of the table, the corresponding number in the third column represents the deduction to be made as a correction for the necessary excess of soap solution. Then the remaining number of test measures, divided by two, gives the hardness of the water in degrees.

Degrees of hardness.	Test measures of soap solution.	Differences for the successive degrees of hardness.
Pure water	0·9	
1°	2·9	..... 2·0
2	5·4	..... 2·5
3	7·7	..... 2·3
4	9·9	..... 2·2
5	12·0	..... 2·1
6	14·0	..... 2·0
7	16·0	..... 2·0
8	18·0	..... 2·0
9	20·0	..... 2·0
10	22·0	..... 2·0
11	24·0	..... 2·0
12	26·0	..... 2·0
13	28·0	..... 2·0
14	30·0	..... 2·0
15	32·0	..... 2·0
16	34·0	..... 2·0

When the water requires more solution of soap to produce a froth, than the standard to which it approximates, and less than the next higher standard, the fraction of a

degree is estimated from the difference between the quantities of soap solution required by the two standards. If, for instance, this difference for the standards s and $s+1°$ amounts to 2 test measures, and the water under examination requires $\frac{9}{10}$ of a test measure more soap solution than the standard s, the hardness of the water would be s $\frac{45}{100}$, for:

$$2 : \tfrac{9}{10} = 1 : \tfrac{45}{100}.$$

When the amount of calcareous salts in water is more than equivalent to sixteen grains of carbonate of lime in the gallon, the degree of hardness indicated by the soap test does not correspond with the amount of calcareous salts in the water, and, in this case, when the water is mixed with pure water, the degree of hardness referable to the water is found to be greater than that indicated by the direct application of the soap test. The difference has been termed the *latent hardness*, and is that hardness which arises from the presence of a larger amount of calcareous salts, than is equivalent to sixteen grains of carbonate of lime in the gallon, and also from the presence of magnesian salts in almost all instances.

In order to estimate this latent hardness, the water is mixed with one or more volumes of pure water, according to the degree of hardness indicated in the first experiment, and the soap test applied in the usual manner to 1000 grain measures of the mixture. The degree of hardness thus found is multiplied by the number expressing the volumes of pure water added to the water under examination, and the result compared with that of the original experiment; the difference expresses the latent hardness.

Supposing the degree of hardness indicated in the first instance to be 24°·6, and that found in the second experiment, after the water had been mixed with an equal volume of pure water, to be 13°·2, the latent hardness of this water would be 2°·2, for:

$$13°{\cdot}2 \times 2 = 26°{\cdot}4, \text{ and } 26°{\cdot}4 - 24°{\cdot}6 = 2°{\cdot}2.$$

With regard to the hardness of water, there is a difference which always should be attended to in the estimation. It consists in the circumstance that the hardness resulting from the presence of carbonates of lime and magnesia, dissolved by

the aid of carbonic acid, may be almost entirely removed by boiling the water, while that resulting from the presence of other calcareous and magnesian salts is unaffected by this treatment, and is permanent.

It is frequently desirable to ascertain how much of the total hardness of water, H, is temporary hardness, *h*, and how much is permanent hardness, h. For this purpose a quantity of the water is boiled in a flask for an hour, with a long tube fitted into the mouth by a cork, so that no great evaporation can take place. It is then made up to the original volume by adding pure water, allowed to cool, and the hardness estimated in the manner already described. The temporary hardness *h* is found by the difference between the two results—

$$h = H - h.$$

The experiment may also be made by adding lime to the water in such quantity as to decompose the bicarbonate, and precipitate the whole as carbonate.

For this purpose milk of lime is made with some of the water under examination, and gradually added, with occasional stirring, to about a pint of the water until there is a very slight excess of caustic lime, which is indicated by a very slight yellow tint produced when some of the water is added to a solution of nitrate of silver in a white basin. The water is then left exposed to the air for about twenty-four hours, again tested with nitrate of silver, and when no yellow colour appears, the soap test is applied in the usual manner, and the difference between the result and that obtained in the first instance represents the temporary hardness.

The above remarks apply especially to hardness caused by the presence of calcareous salts in water. The hardness of water, resulting from the presence of quantities of magnesian salts equivalent to calcareous salts is indicated by the soap test up to 6°; but above that point, the degree of hardness is not indicated uniformly by the quantity of soap solution required to produce the froth. When, however, calcareous and magnesian salts are associated together in water, the indications of the soap test do not bear any uniform proportion to the amount of these salts present in water.

This remarkable influence, exercised by magnesian salts, is greater in proportion to their amount. Thus, water containing an amount of calcareous salts corresponding to 6°, together with magnesian salts corresponding to 1° hardness, does not require so much soap solution, to produce a froth, as if the magnesian salts were absent, by 0·4 of a test measure; and water containing calcareous and magnesian salts, each in amount corresponding to 16° hardness, gives a froth with only 27·9 test measures of soap solution.

It is therefore necessary, in all instances, after applying the soap test for hardness, to ascertain whether or not magnesia is present in the water.

The presence of magnesian salts in water may be recognized, in applying the soap-test, by the appearance of a curd besides the froth produced by calcareous salts.

When magnesian salts are present together with calcareous salts, it is necessary to make some further experiments for the purpose of ascertaining their several amounts, in the manner described under the estimation of lime and magnesia (p. 249).

In estimating the degree of hardness of water containing magnesian salts, together with calcareous salts, a second experiment should be made after separating lime by oxalate of ammonia, as directed at p. 249. The following table shows the quantity of soap-solution requisite for producing the froth with water containing different amounts of magnesian salts; the degrees of hardness representing quantities of magnesian salts, in a gallon of water, equivalent to 1, 2 or more grains of carbonate of lime :—

Degrees of hardness.	Test-measures of soap-solution.	Degrees of hardness.	Test-measures of soap-solution.
1	3·2	9	16·0
2	5·4	10	16·9
3	7·6	11	17·7
4	9·5	12	18·3
5	11·5	13	18·7
6	13·4	14	19·1
7	14·8	15	19·4
8	15·4	16	19·6

Suspended substance in water is best estimated, though only approximatively, by allowing a large quantity of the

water to settle in a stoppered bottle, then drawing off the clear water, by means of a syphon, and collecting the sediment upon a dry, weighed filter, which is again dried and weighed.

In the analysis of mineral water it is sometimes desirable to test for, and estimate, some substances that are present only in very small amount. For this purpose a considerable quantity of the water should be evaporated nearly to dryness, the residue extracted with alcohol of 85 per cent., the undissolved portion separated by filtration, washed with alcohol, the alcoholic filtrate evaporated to dryness, and redissolved in water. Both this solution and the insoluble residue are preserved for further examination.

B. Methods of effecting solution.

The reagents chiefly used as solvents in the wet way, are hydrochloric acid, sulphuric acid, carbonated or caustic alkali, and hydrofluoric acid or fluoride of ammonium.

Some substances which resist solution in the wet way, either wholly or partially, must be rendered soluble by fusion with an alkali.

Very few substances of this class are soluble to any appreciable extent in water. It is only soils that contain small proportions of substances, which are soluble in water, and which it is important to examine separately, because there is much reason for the opinion that the immediate value of a soil is in great measure dependent upon the solubility of the mineral substances required for the growth of any particular crop.

Solution by water. In order to extract from a soil the substance soluble in water, a considerable quantity (5 or 10 pounds) of the air-dried, sifted soil is mixed with a known quantity of water. The mixture is frequently shaken, and digested during several days, then allowed to settle, and a quantity of the perfectly clear liquid, equal to one half of the water used, drawn off by a siphon into a porcelain basin, evaporated to about $\frac{1}{30}$th its volume, transferred to a weighed platinum capsule, and evaporated to dryness at 212° Fahr. This residue will contain the soluble substance of half the quantity of soil operated upon.

The result thus obtained is sufficiently accurate for all practical purposes. The complete extraction of all the soluble substance would be an almost endless task, particularly when the soil contained gypsum, and the value of the result would bear no proportion to the amount of time and labour expended.

Solution by hydrochloric acid. The reagent most generally applicable for the solution of these substances is hydrochloric acid, by reason both of its powerful electronegative character and of the general solubility of the chlorides. In some instances its decomposing action may be increased by the addition of a few drops of nitric acid.

In the analysis of soils, the residue left after estimating the organic substance (p. 223) is used for extracting the portion soluble in hydrochloric acid. It should be well mixed, and 20 grms. taken for the experiment, mixed with water in a flask, so as to form a thin paste, hydrochloric acid added gradually, and the whole digested for two hours at a temperature of 200° Fahr. Water is then added, the undissolved portion separated by filtration, washed, and the filtrate evaporated, or made up by dilution to a convenient volume, preserved for analysis in a stoppered bottle labeled "*hydrochloric solution.*"

Other substances are treated in the same manner, but a smaller quantity, from 1 to 3 grms., is sufficient. When owing to the presence of carbonate in large proportion, the effervescence is violent, it must be allowed to subside before adding more acid, and the flask should be slightly inclined, so that particles of the liquid may not be thrown out by the gas evolved.

Ashes of plants sometimes leave a residue insoluble in hydrochloric acid, even with addition of a few drops of nitric acid. Such a residue is to be collected on a filter, washed, dried at 212° Fahr., and weighed. As much as possible of it is then shaken into a silver basin, boiled with caustic soda, water added, and the liquid filtered through the same filter. Any residue that may still remain undissolved will consist of sand and charcoal. It is dried and weighed, ignited and again weighed. The last weighing gives the amount of sand and the difference between the two—that of charcoal.

Solution by sulphuric acid. In the analysis of soils, marl, &c., the argillaceous portion which is not dissolved by hydrochloric acid, may often be conveniently rendered soluble by treatment with concentrated sulphuric acid. For this purpose a weighed quantity of the residue left from the previous operation is covered with the acid in a platinum basin and heated, in a place where the fumes will be carried away, until a dry residue remains. This is heated with dilute hydrochloric acid, the solution filtered, and set aside for analysis in the same manner as the other solutions, in a stoppered bottle labeled "*sulphuric solution.*"

Solution by fusion with alkali. It generally happens that a portion of soils, clay, marl, &c., altogether resists the solvent action of hydrochloric or sulphuric acids, and some substances may be regarded as insoluble. The most perfect kinds of glass do not wholly resist the solvent action of strong acids or boiling alkaline liquids, but the amount dissolved is too small to admit of the use of these reagents for the purposes of analysis.

These insoluble substances are for the most part silicates. They may all be rendered soluble by fusion with an excess of alkali. For this purpose from 1 to 3 grms. of the finely powdered, dry substance is mixed with three or four times its weight of anhydrous carbonated alkali (p. 32) in a platinum crucible, which is then covered with a lid with a small hole in the centre, and exposed to a bright red heat for about three quarters of an hour. When cold, the crucible with its contents is placed in water, in a beaker glass covered with a short-necked funnel, through which, when the solution is sufficiently advanced, hydrochloric acid is gradually added until in excess. The crucible is then taken out of the liquid, laid upon the funnel, and washed to remove adherent portions. The solution is set aside in the beaker labeled "*solution by fusion with alkali.*"

Solution by hydrofluoric acid or *fluoride of ammonium.* The silicates which require to be fused with an alkali, sometimes contain, besides other bases, alkalies; and when it is requisite to estimate these directly, hydrofluoric acid must be employed for solution. The best method of effecting solution by means of this reagent is to expose the finely powdered substance, covered with water in a platinum capsule,

to an atmosphere of the gas, generated by heating a mixture of fluorite and sulphuric acid. The apparatus required (fig. 63) is a leaden chamber about eight inches wide and four inches deep, with a closely fitting lid and a small tripod stand for supporting the platinum capsule above the mixture of fluorite and acid which is spread upon the bottom of the chamber.

Fig. 63.

The action of the hydrofluoric acid may be facilitated by moistening the substance with concentrated solution of ammonia, which absorbs the gas more readily than water, while the resulting fluoride of ammonium acts upon the silicates.

When the substance is perfectly decomposed, there is no longer any gritty sound when a platinum rod is rubbed upon the bottom of the capsule; excess of concentrated sulphuric acid is then added, the liquid evaporated to dryness, the residue ignited, until vapour is no longer evolved, then moistened with concentrated hydrochloric acid, and, after some time, dissolved in water.

When the decomposition of the silicate has been complete, the residue left, after evaporation to dryness, should be completely soluble in water; but when the amount of lime in the silicate is large, a considerable quantity of water will be requisite, because the lime will be in the state of sulphate. The solution is made up to a convenient volume, and preserved for analysis in a bottle labeled "*solution by hydrofluoric acid.*"

Solution by fusion with bisulphate of potash.—The compounds of alumina with bases are sometimes rendered soluble by fusion with bisulphate of potash, more readily than by any other reagent. The substance for analysis is mixed with six parts of bisulphate and heated in a platinum crucible until the melted mass becomes clear and is no longer agitated. This, when cold, is treated with water, and any insoluble portion separated by filtration; the filtrate being preserved for analysis in a stoppered bottle labeled "*solution by fusion with bisulphate of potash.*"

Sometimes it is only necessary to ascertain the total amount of the several constituents of the substance under

examination; as in the analysis of ashes of plants, or of substances that are chemically homogeneous, such as glass, and then, that reagent is selected for effecting solution which is found best to answer the purpose. When, however, a substance is partly dissolved by *several* reagents, it may be desirable to ascertain the chemical nature of the several portions. This is often the case in the analysis of mixed substances, of some minerals, and more particularly so in the analysis of soils.

The constituents of a substance under examination may therefore be contained in one or more solutions:—

		In the analysis of
a.	*Water solution*	Soils and water.
b.	*Hydrochloric solution*	Soils, marl, limestone, &c.
c.	*Sulphuric solution*.....	Soils, clay, &c.
d.	*Hydrofluoric solution*	Alkaline and other silicates.
e.	*Solution obtained by fusion*	
	ϵ { *with alkaline carbonates* }	Siliceous minerals, &c.
	η { *with bisulphate of potash* }	Aluminous minerals.

When a substance is dissolved by more than one solvent, the portion left undissolved by each, should always be washed until all soluble substance is removed. The dried residue is to be weighed, for the purpose of obtaining a control for the results of the analysis, and the washings should be mixed with the filtrate.

In each instance great care should be taken to avoid loss in filtration, and, in the subsequent steps of the analysis, it is generally advisable to operate upon a portion only of any solution; so that in case an experiment fails, or there is need of repeating an analysis, or of applying qualitative tests, the remainder of the solution may be employed. The analytical results obtained from such known fractions of a solution, must be correspondingly multiplied, in order that they may bear a proper relation to the amount of substance operated upon in the first instance.

For the purpose of separating a solution into fractional parts, it should be collected in a flask, with a narrow neck

bearing a mark indicating some convenient volume, to which the solution is made up by dilution, or by evaporation. The fractional parts are best measured by means of pipettes gauged to deliver known volumes related to that of the solution.

C. Actual analysis.

In the following description of methods of separation, they are so arranged, that under the head of each constituent, those are placed first, which are adapted for its separation from individual other constituents of the substances here referred to, while those which are adapted for its separation from several others, follow after.

When the substance to be analysed is known to consist of two or more constituents, for the separation of which a special method is given, the solution of the substance, by the methods already described, will, in many instances, be unnecessary.

It is advisable, at each stage of the analysis, to label the funnels and beakers containing the precipitates and filtrates, so that no error may arise from their confusion.

Examination of the water solution.

The only cases in which a water solution would be obtained, are, the analysis of soils, and of natural water; and the estimation of chlorine in ashes. The substances which, after the preliminary treatment (p. 200), may be present in the water solution, and in any considerable amount, are chlorine, sulphuric, carbonic, (phosphoric) and nitric acids; (silica), (alumina), oxides of iron, lime, magnesia, and alkalies.

The water solution from soils may, owing to the presence of organic substance, contain the substances indicated by brackets in larger amounts than would otherwise be dissolved by water.

Total amount of solid substance.—This, in the case of water, or of the water solution from a soil, is estimated by evaporating to dryness, on a steam bath, a known volume of the liquid and weighing the residue well dried at 212° F.

The quantity of water or solution to be evaporated should

be either a gallon, 10·000 grains, or less, according to the amount of dissolved substance.

The evaporation should be conducted in a platinum capsule very thin and light, for convenience in weighing. It need not be large enough to hold the whole of the liquid, which may be added as the evaporation proceeds.

Fixed mineral substance; estimated by igniting the residue obtained in the last experiment; when organic substance is present, maintaining the heat until every trace of carbon is burnt, and again weighing. The loss of weight by ignition may represent organic substance, ammoniacal salts, nitric acid, and sometimes a certain amount of water. Loss of carbonic acid, in the case of carbonates and organic substance being associated in the water solution, must be guarded against in the manner described at page 223.

Chlorine; estimated by precipitation as chloride of silver in a part of the solution acidulated with nitric acid.

The experiment may be made volumetrically with a silver solution of known value (p. 180); or the chloride of silver may be collected and weighed (p. 153).

In the latter case, the quantity, x, of chlorine is calculated from the quantity, q, of chloride obtained, according to the proportion—

$$x : q = \mathrm{Cl} : \mathrm{AgCl}.$$

The estimation of chlorine in the ashes of plants, &c. is made by special experiment with the water solution from a known quantity of ash.

Sulphuric acid; estimated by precipitation as sulphate of baryta in a part of the solution.

Sulphuric acid may be separated as sulphate of baryta from all acids and bases with which it may be associated in solution.

The solution is mixed with excess of hydrochloric acid, chloride of barium solution added until no further precipitate is separated, the liquid kept hot until the precipitate has perfectly subsided, the clear portion decanted and filtered; the precipitate is then warmed with a very dilute solution of chloride of ammonium, collected upon the filter, washed with hot water containing some chloride of ammonium, dried, ignited, and weighed.

The quantity x of [illegible] quantity, q, of [illegible] proportion :—

$$x : q = [illegible]$$

Sometimes the [illegible] tically by the [illegible]

Carbonic acid: [illegible] rated to a [illegible] apparatus fig. [illegible] The loss of weight [illegible]

Nitric acid. A portion of the [illegible] evaporating a [illegible] or the water solution if a soluble [illegible] volume of concentrated sulphuric acid and [illegible] with a solution of protosulphate of iron. If any nitric acid is present, the liquid immediately becomes more or less almost black or, when the quantity of nitric acid is very minute, reddish brown. The colour disappears when the liquid is boiled.

In order to estimate the nitric acid in water, a quantity of the mother liquor [illegible] representing a known amount of the water, is mixed with solution of sulphate of baryta until no further precipitate is produced. The mixture is then separated by filtration. The filtrate evaporated to a small bulk, in a small retort, the residue mixed with sulphuric acid, in considerable excess, and the nitric acid distilled over into baryta water. The excess of baryta is then precipitated as carbonate by a stream of carbonic acid, the liquid evaporated to dryness, on a sand-bath, the residue treated with water, and the undissolved substance separated by filtration. The clear filtrate is then evaporated to dryness, the residue weighed, and the quantity of nitric acid calculated from q, that of nitrate of baryta obtained, by the proportion—

$$x : q = NO^5 : BaO,NO^5.$$

Silica. The residue of fixed mineral substances [illegible] is moistened with hydrochloric acid, and after standing for an hour, dissolved in water, evaporated to dryness, and silica separated as in the case of the hydrochloric solution.

be either a gallon, 10·000 grains, or less, according to the amount of dissolved substance.

The evaporation should be conducted in a platinum capsule very thin and light, for convenience in weighing. It need not be large enough to hold the whole of the liquid, which may be added as the evaporation proceeds.

Fixed mineral substance; estimated by igniting the residue obtained in the last experiment; when organic substance is present, maintaining the heat until every trace of carbon is burnt, and again weighing. The loss of weight by ignition may represent organic substance, ammoniacal salts, nitric acid, and sometimes a certain amount of water. Loss of carbonic acid, in the case of carbonates and organic substance being associated in the water solution, must be guarded against in the manner described at page 223.

Chlorine; estimated by precipitation as chloride of silver in a part of the solution acidulated with nitric acid.

The experiment may be made volumetrically with a silver solution of known value (p. 180); or the chloride of silver may be collected and weighed (p. 153).

In the latter case, the quantity, x, of chlorine is calculated from the quantity, q, of chloride obtained, according to the proportion—

$$x : q = \mathrm{Cl} : \mathrm{AgCl}.$$

The estimation of chlorine in the ashes of plants, &c. is made by special experiment with the water solution from a known quantity of ash.

Sulphuric acid; estimated by precipitation as sulphate of baryta in a part of the solution.

Sulphuric acid may be separated as sulphate of baryta from all acids and bases with which it may be associated in solution.

The solution is mixed with excess of hydrochloric acid, chloride of barium solution added until no further precipitate is separated, the liquid kept hot until the precipitate has perfectly subsided, the clear portion decanted and filtered; the precipitate is then warmed with a very dilute solution of chloride of ammonium, collected upon the filter, washed with hot water containing some chloride of ammonium, dried, ignited, and weighed.

The quantity x of sulphuric acid is calculated from the quantity, q, of sulphate of baryta obtained, according to the proportion:—

$$x : q = SO^3 : BaO\,SO^3.$$

Sometimes the sulphuric acid may be estimated volumetrically by the method described at page 79.

Carbonic acid; estimated in a part of the solution evaporated to a small bulk, and treated with sulphuric acid in the apparatus fig. 20, and in the manner described at page 66. The loss of weight, c, gives the amount of carbonic acid.

Nitric acid. A portion of the mother liquor, obtained by evaporating a considerable quantity of the water (p. 222), or the water solution of a soil, is mixed with an equal volume of concentrated sulphuric acid, and, when cold, with a solution of protosulphate of iron. When nitric acid is present, the liquid immediately becomes dark-coloured, almost black, or, when the quantity of nitric acid is very minute, reddish brown. The colour disappears when the liquid is boiled.

In order to estimate the nitric acid in water, a quantity of the mother liquor (p. 230), representing a known quantity of the water, is mixed with solution of sulphate of silver, until no further precipitate is produced, the chloride of silver separated by filtration, the filtrate evaporated to a small bulk, in a small retort, the residue mixed with sulphuric acid, in considerable excess, and the nitric acid distilled over into baryta water. The excess of baryta is then precipitated as carbonate by a stream of carbonic acid, the liquid evaporated to dryness, on a steam-bath, the residue digested with water, and the undissolved substance separated by filtration. The clear filtrate is then evaporated to dryness, the residue weighed, and the quantity of nitric acid, x, calculated from q, that of nitrate of baryta obtained, by the proportion—

$$x : q = NO^5 : BaO,NO^5.$$

Silica. The residue of fixed mineral substance (p. 236), is moistened with hydrochloric acid, and, after standing for an hour, dissolved in water, evaporated to dryness, and silica separated as in the case of the hydrochloric solution.

Phosphoric acid; tested for in the hydrochloric acid solution of a portion of the deposit obtained by evaporating a considerable quantity of the water (p. 222), as directed at p. 239, and estimated, as in the hydrochloric solution, according to the nature of the substances with which it may be associated.

Bases are estimated, in the filtrate from silica, in the same manner as in the hydrochloric acid solution.

Iodine; tested for, in the solution obtained by means of alcohol from the residue left by evaporating a large quantity of water (p. 230), either with starch and chlorine water (p. 182), or with nitrate of palladium, by means of which also, it would be separated and estimated as iodide of palladium. The quantity, x, is calculated from q, that of the iodide of palladium obtained, by the proportion—

$$x : q = \mathrm{I} : \mathrm{PdI}.$$

Bromine; tested for in another portion of the solution, or, when iodine is present, in the filtrate from iodide of palladium, with chlorine water and ether which dissolves the liberated bromine and acquires a yellow colour. The ether solution may be mixed with caustic potash, evaporated to dryness, the residue ignited and tested with binoxide of manganese and sulphuric acid (p. 181). Bromine is separated from chlorine and estimated in the filtrate from iodide of palladium, as directed at p. 185.

Ammonia; estimated by evaporating a considerable quantity of the water, mixed with excess of hydrochloric acid, to a small bulk, and distilling the residue with caustic soda, into dilute hydrochloric acid, evaporating the distillate nearly to dryness, and precipitating the ammonia as chloride of platinum and ammonium, with bichloride of platinum, as directed at p. 199. The quantity, x, is calculated from q, that of the chloride of platinum and ammonium obtained, by the proportion—

$$x : q = \mathrm{NH^3} : \mathrm{PtCl^2,NH^4Cl}.$$

Examination of the hydrochloric solution.

Silica is almost always present. It is always separated, before any other constituent.

1. *Separation of silica from all the bases.*—The whole of the solution is evaporated to dryness, in a porcelain basin at about 200° F. The residue is kept hot for some time, to ensure the perfect separation of water; then moistened with hydrochloric acid, and after about an hour heated with dilute hydrochloric acid enough to dissolve the bases, &c. present. The silica, remaining as a white powder, is collected upon a filter, washed, thoroughly dried, ignited, and weighed.—The filtrate, *f.* 1, contains all the other constituents.

In some instances of decomposition of siliceous substances by hydrochloric acid, the silica is only partially dissolved, the greater part remaining in a gelatinous or pulverulent state, and then great care must be taken to prevent loss by spirting during the evaporation to dryness.

It is frequently desirable to test the silica thus separated, by dissolving it by hydrofluoric acid and evaporating the solution to dryness (p. 232), in order to ascertain whether the bases have been perfectly separated.

Sulphuric acid is tested for, and estimated, in a part of the filtrate from the silica, as in the water solution, p. 236.

Phosphoric acid is very frequently present, and is generally associated with bases which form with it compounds insoluble in water.

In the analysis of water, limestone, clay, &c., the search for phosphoric acid may generally be omitted altogether; for though seldom absent, its amount is inconsiderable.

In the analysis of soils, ashes, &c., it is necessary to test for phosphoric acid; not only because its amount is one of the most important data of some analyses, but also, because the method of analysis to be adopted depends much upon the presence or absence of this substance.

The best test for phosphoric acid is a solution of molybdate of ammonia, mixed with excess of nitric acid: it produces a yellow precipitate in acid solutions containing phosphoric acid. This test is applied to a portion of the filtrate from silica, previously concentrated by evaporation.

Phosphoric acid is estimated as pyrophosphate of magnesia, $2MgO,PO^5$, obtained by igniting the phosphate of magnesia and ammonia, $2MgO,NH^4O,PO^5$.

When the solution does not contain any bases that would be precipitated as insoluble phosphates by ammonia, and when they have been separated, the solution is mixed with excess of ammonia, or, if alkaline, neutralized with hydrochloric acid, and then mixed with excess of ammonia. Solution of sulphate of magnesia, mixed with as much chloride of ammonium as will prevent precipitation of magnesia by ammonia, is then added; the whole kept for two hours at a temperature not higher than 86° F., the precipitate collected by filtration, washed with hot ammoniacal water, dried, ignited, and weighed.

The quantity, x, of phosphoric acid, is calculated from the quantity, q, of pyrophosphate obtained, according to the proportion:—

$$x : q = PO^5 : 2MgO,PO^5.$$

For the precipitation of phosphoric acid in this state it is essential that the solution should not contain any bases that would be precipitated as insoluble phosphates by ammonia. The first step in the process, therefore, is the separation of phosphoric acid from such bases as form with it salts that are not soluble in water, for instance oxides of iron and manganese, alumina, lime, magnesia, &c.; the mode of effecting this separation varies according to circumstances as follows.

2. *Separation of phosphoric acid from magnesia.*—Phosphate of magnesia is precipitated from the filtrate, *f.* 1, by adding ammonia in very slight excess, the precipitate collected upon a filter, washed, dried, ignited, and weighed.—It is then dissolved by nitric acid, mixed with mercury, in such quantity that a small globule remains undissolved, while the whole is evaporated to complete dryness upon a steam bath. When the residue smells of nitric acid, it must be moistened with water, and again evaporated to dryness, this operation being repeated until the dry residue does not smell of nitric acid while still warm. It is then covered with hot water, transferred to a very small filter, and washed with hot water until a drop of the filtrate, *f.* 2, evaporated to dryness, leaves no residue that is not volatilized at a red heat. The washed residue—containing the whole of the phosphoric acid combined with suboxide of mercury—is thoroughly dried, transferred to a platinum crucible, mixed with carbonate of soda and potash, the filter laid upon the top, and covered with

carbonate of soda and potash. The crucible is then moderately heated under an open flue, for half an hour, and when vapour is no longer given off, the temperature gradually raised to redness. The melted mass is dissolved in water, and phosphoric acid precipitated, as phosphate of magnesia and ammonia, from the solution, as above directed.

3. *Separation of phosphoric acid from lime.*—The solution, which should not be very dilute, is mixed with twice its volume of strong alcohol, and sulphuric acid added so long as any precipitate is produced. The precipitate, *p.* 1, of sulphate of lime, which is quite insoluble in alcohol, is separated by filtration, and washed with alcohol.—After evaporating the alcohol from the filtrate, phosphoric acid is precipitated, as phosphate of magnesia and ammonia, as above directed.

4. *Separation of phosphoric acid from alumina.*—Phosphate of alumina is precipitated from the filtrate, *f.* 1, by adding ammonia in very slight excess, the precipitate collected upon a filter, washed, dried, ignited, and weighed.—This precipitate, mixed with three-fourths its weight of silica, and six times its weight of carbonate of potash and soda, is heated to redness for half an hour in a platinum crucible, the ignited mass digested with water, the insoluble portion, *p.* 2, separated by filtration, and washed. The solution—containing the whole of the phosphoric acid, in the state of phosphate of soda—is mixed with hydrochloric acid in excess, evaporated to complete dryness on a steam bath, the residue digested with water, the silica separated by filtration, and phosphoric acid precipitated, as phosphate of magnesia and ammonia, from the filtrate, as above directed.

5. *Separation of phosphoric acid from oxides of iron, or manganese.*—The phosphate of iron or manganese is precipitated from the filtrate, *f.* 1, by adding ammonia in very slight excess, the precipitate collected upon a filter, washed, dried, ignited, and weighed; then melted with twice its weight of carbonate of soda and potash. The melted mass is digested with water, the insoluble residue, *p.* 3, separated by filtration, and washed. Phosphoric acid is precipitated, as phosphate of magnesia and ammonia, from the filtrate, as above directed.

When the solution contains compounds of calcium, magnesium, manganese, or aluminum together with iron, and when,

as is generally the case in the analysis of soils, the amount of iron is proportionately much larger than that of phosphoric acid, the following method of separation may be adopted.

6. The solution is mixed with ammonia until there begins to be a precipitate that does not redissolve when the liquid is stirred. It is then mixed with acetic acid until strongly acid, and, with acetate of soda until it becomes red; showing that the perchloride of iron has been converted into acetate of iron. The liquid is then boiled until the colour disappears, and a brown precipitate, *p.* 4, is formed, owing to the decomposition of the peracetate of iron. This precipitate—containing all the phosphoric acid—is collected upon a filter and washed. The phosphoric acid may be separated from the oxide of iron, &c., by fusion with silica and carbonate of potash and soda, as above directed.

When the amount of iron is proportionately very large, the separation of phosphoric acid by the method just described, is attended with some inconvenience, and the following modification of this method is preferable, perhaps in most instances.

7. The solution is heated to the boiling-point, mixed with sulphite of soda, until the colour becomes pale green, and a white precipitate is produced by carbonate of soda; it is then boiled until the smell of sulphurous acid is no longer recognizable, and any excess of acid neutralized by a few drops of carbonate of soda solution.—The liquid is then mixed with a few drops of chlorine water and excess of acetate of soda. More chlorine water is then added, very gradually, until the liquid begins to acquire a distinct red colour; it is then boiled until clear, the precipitate, *p.* 5, quickly collected by filtration, and washed with hot water. This precipitate, as in the previous case, contains all the phosphoric acid, and is treated in the same way for the estimation of the acid.

8. When the solution does not contain enough iron for the separation of the whole of the phosphoric acid by this method, perchloride of iron may be added until the liquid presents a decided red colour on the addition of acetate of soda, and the phosphoric acid then precipitated, as basic perphosphate, as above directed.

9. Or, phosphoric acid may be partially precipitated by

means of acetate of soda, as perphosphate (Fe^2O^3,PO^5). This precipitate, *p.* 6, is collected upon a filter, washed, and the remainder of the phosphoric acid precipitated by adding perchloride of iron to the filtrate, and operating in the manner already described.

Iron; estimated in a part of the filtrate, *f.* 1, from silica, as peroxide (Fe^2O^3), which is precipitated by ammonia.

The liquid is heated to the boiling-point, and mixed with ammonia until there is a very slight excess; after the precipitate has subsided, the clear liquid is poured through a filter, the precipitate washed several times with boiling water, by decantation, then collected upon the filter, thoroughly washed with boiling water, dried, ignited, and weighed.

The quantity, x, of iron, is calculated from the quantity, q, of peroxide obtained, by the proportion:—

$$x : q = Fe : Fe^2O^3.$$

When the iron is in a state of combination corresponding to FeO, the liquid must be boiled with nitric acid, in order to convert the iron into a state of combination corresponding to Fe^2O^3, before adding ammonia.

10. *Separation of iron from potassium or sodium.*—When the solution does not contain, besides iron, compounds of any other metals than potassium or sodium, the precipitation, by means of ammonia, may be effected at once and without any special precautions.

11. *Separation of iron from magnesium and calcium.*—When the solution contains compounds of magnesium or calcium, the separation of iron may be effected in the same manner, but the greatest care must be taken to avoid the introduction of carbonic acid, either as an impurity in the ammonia used, or, by absorption from the atmosphere, since in that case carbonate of magnesia or lime would be precipitated together with the peroxide of iron.

12. *Separation of iron from aluminum.*—The solution, containing iron in a state of combination corresponding to Fe^2O^3, is mixed with a considerable excess of caustic potash or soda, boiled for ten minutes in a silver basin, diluted with water, the precipitate collected upon a filter, washed, dissolved by hydrochloric acid, and iron precipitated, as peroxide, from the solution, as above directed.

When the oxide of iron amounts to more than $\frac{1}{100}$ of the alumina, the treatment with potash requires to be repeated several times, in order to effect complete separation.—In such a case the following method of separation is better.

13. Oxide of iron and alumina are precipitated together from a part of the filtrate, *f.* 1, by means of ammonia, collected upon a filter, washed, dried, ignited, and weighed. A convenient portion of the finely powdered mixture is weighed in a porcelain tray, heated to redness for some time in a tube, through which a current of dry hydrogen is passed. By this means the oxide of iron is reduced to the state of metal. When the tube is cold, the tray is taken out, the contents washed into a beaker, and digested with dilute nitric acid. The iron dissolves, and, after separating the white residue of alumina, *p.* 7, by filtration, is precipitated, as peroxide of iron, from the filtrate, as above directed.

14. *Separation of iron from manganese.*—A part of the filtrate, *f.* 1, is nearly neutralized with ammonia, the iron precipitated as basic succinate of iron, by means of solution of succinate of ammonia or of succinate of soda, the precipitate collected upon a filter, washed with *cold* water, then with ammonia, to remove the succinic acid, and the residual peroxide of iron dried, ignited, and weighed.

15. When the solution does not contain phosphoric acid, but contains besides iron, compounds of aluminum, together with compounds of calcium or magnesium, a portion of the filtrate, *f.* 1, is heated to the boiling point, mixed with chloride of ammonium and a very slight excess of ammonia, or sulphide of ammonium, the precipitate, consisting of sesquioxide or sulphide of iron and alumina, collected upon a filter, with the precautions mentioned in 11, washed and treated as directed in 12, 13, or 18.

16. When the solution does not contain phosphoric acid, but contains manganese together with compounds of calcium or magnesium, either with or without alumina, a portion of the filtrate, *f.* 1, is saturated with chlorine, mixed with a very slight excess of ammonia, the precipitate separated by filtration, with the precautions mentioned in 11, washed, dissolved by hydrochloric acid, and the solution treated as directed in 26 for the separation of manganese, the precipitate dissolved by hydrochloric acid, baryta sepa-

rated as sulphate from the solution, and the iron separated from alumina as directed in 12 or 13.

17. In either of the two previous cases the solution may be nearly neutralized with soda, and digested for twelve hours with excess of carbonate of baryta; the precipitate—containing the sesquioxide of iron, alumina, and, in the latter case, sesquioxide of manganese, if the solution was mixed with chlorine water—washed, dissolved by hydrochloric acid, the solution boiled, manganese separated as directed in 24, the precipitate produced by carbonate of baryta dissolved by hydrochloric acid, baryta separated as sulphate, and the filtrate treated as directed in 12.

18. Sulphide of ammonium may also be used as the precipitant in either case, the washed precipitate should then be dissolved by hydrochloric acid, the solution boiled with nitric acid, and iron separated from alumina as in 12 or 13; or, when the solution contained manganese, the solution is first treated as in 24, for the separation of manganese.

19. When the solution contained phosphoric acid, together with compounds of magnesium or calcium, and the phosphoric acid has been separated as basic perphosphate of iron —6 or 7—the residue, *p.* 4, *p.* 5, or *p.* 6, is dissolved by hydrochloric acid, and iron precipitated from the solution, as peroxide, by means of ammonia, as above directed.

20. When the solution contained compounds of aluminum, together with phosphoric acid, either with or without compounds of calcium or magnesium, the residue, *p.* 2, left after separating the phosphoric acid from the basic perphosphate, by fusion with silica and alkaline carbonate, is dissolved by hydrochloric acid, the solution mixed with the filtrate from the perphosphate, evaporated to dryness, as above directed, to separate silica, and the filtrate treated as in 15.

21. When the solution contained manganese, together with phosphoric acid, either with or without compounds of aluminum, calcium, or magnesium, the residue, *p.* 3, left after separating phosphoric acid, and the filtrate from the basic perphosphate are treated as directed in 20, and the filtrate, *f.* 3, from silica is treated as directed in 16, 17, or 18, according to the nature of the substances present in solution.

Manganese; estimated as oxide (Mn^3O^4), which is obtained by igniting hydrated protoxide, sesquioxide, or carbonate.

For the precipitation of carbonate of manganese the liquid is heated to the boiling-point, mixed with carbonate of soda in excess, and the heat maintained for an hour; the precipitate is collected upon a filter, washed, dried, and ignited.

For the precipitation of hydrated protoxide of manganese a solution of caustic alkali is used in the same manner.

When the solution contains ammoniacal salts, it must, in both cases, be evaporated to dryness, with excess of the precipitant, so as to ensure their separation.

The quantity, x, of protoxide, or, x', of sesquioxide of manganese, is calculated from, q, that of the red oxide obtained, by the proportions:—

$x : q = MnO : Mn^3O^4$; or, $x' : q = Mn^2O^3 : Mn^3O^4$.

22. *Separation of manganese from magnesium.*—The solution is saturated with chlorine, to convert the manganese into sesquichloride, and hydrated sesquioxide of manganese precipitated by a solution of bicarbonate of potash, the precipitate collected upon a filter, washed, dried, ignited, and weighed.

23. *Separation of manganese from calcium.*—The solution is saturated with chlorine, hydrated sesquioxide of manganese precipitated by ammonia, the precipitate rapidly separated by filtration, washed, dissolved by hydrochloric acid, again precipitated, as hydrated sesquioxide, washed, dried, ignited, and weighed.

24. *Separation of manganese from aluminum.*—The solution is boiled with hydrochloric acid, nearly neutralized with carbonate of soda, when cold digested with excess of carbonate of baryta for twelve hours, and frequently stirred meanwhile. The precipitate is separated by filtration, baryta separated from the filtrate as sulphate, and manganese precipitated as carbonate, as above directed.

25. When the solution does not contain phosphoric acid, or compounds of any other metals besides iron, manganese is estimated in the filtrate from basic succinate of iron (14) by precipitation as carbonate, as above directed.

26. When the solution does not contain phosphoric acid, but contains compounds of iron and aluminum, a part of the filtrate, *f.* 1, is boiled with nitric acid, so as to convert the iron into a state of combination corresponding with Fe^2O^3, then with hydrochloric acid to convert the manganese into a state of combination corresponding with MnO, then nearly

saturated with soda, and when cold, digested for twelve hours with excess of carbonate of baryta, the precipitate separated by filtration, washed, baryta separated from the filtrate as sulphate, and manganese precipitated as carbonate of manganese, as above directed.

27. When the solution does not contain phosphoric acid, but contains compounds of calcium or magnesium, together with compounds of iron or aluminum, a part of the filtrate, *f.* 1, is treated as in 16, 17, or 18, manganese separated by means of carbonate of baryta from iron or alumina (26), and estimated as carbonate of manganese, as above directed.

28. When the solution contained phosphoric acid, the residue, *p.* 3, left after fusion with alkaline carbonate, is dissolved by hydrochloric acid, and manganese precipitated as carbonate, as above directed.

29. When the solution contained phosphoric acid and compounds of iron or aluminum, together with compounds of calcium or magnesium, and the phosphoric acid has been separated as basic perphosphate of iron (6 or 7), the residue, *p.* 4 or *p.* 5, and the filtrate from the basic perphosphate are treated as in 21, and the filtrate, *f.* 3, treated as in 16, 17, 18, or 27.

Aluminum; estimated as alumina, which is precipitated by ammonia or sulphide of ammonium.

The solution is mixed with chloride of ammonium, heated to the boiling point, and sulphide of ammonium or ammonia added until there is a very slight excess; the precipitate is washed two or three times with boiling water, by decantation, collected upon a filter, washed, well dried, ignited, and weighed.

When the solution contains sulphuric acid, the washed precipitate of alumina must be dissolved by hydrochloric acid, and again precipitated by ammonia in order to ensure the separation of sulphuric acid.

30. *Separation of aluminum from potassium or sodium.*—When the solution does not contain, besides alumina, compounds of any other metals than potassium or sodium, the precipitation, by means of ammonia or sulphide of ammonium, may be effected at once, and without any special precaution.

31. *Separation of aluminum from magnesium.*—The solution is nearly saturated with soda and digested with excess of

carbonate of baryta for twelve hours. The precipitate containing the whole of the alumina—is washed, dissolved by hydrochloric acid, baryta separated from the solution as sulphate of baryta, and alumina precipitated by sulphide of ammonium, washed, dried, ignited, and weighed; or,

32. The alumina is precipitated by a solution of bicarbonate of potash, the precipitate washed with cold water, dissolved by hydrochloric acid, alumina precipitated by sulphide of ammonium, washed, dried, ignited, and weighed.

33. *Separation of aluminum from calcium.*—When the solution contains compounds of magnesium or calcium, the precipitation of alumina may be effected by ammonia, but the greatest care must be taken to avoid the introduction of carbonic acid, either as an impurity in the ammonia used, or by absorption from the atmosphere, since in that case, some carbonate of lime would be precipitated. The alumina should always be dissolved by hydrochloric acid, and precipitated again in order to separate a small quantity of lime which is always precipitated in combination with the alumina, in the first instance.

34. Alumina may also be separated from lime by digesting the solution with carbonate of baryta in the same way that it is separated from magnesia (31).

35. When the solution does not contain phosphoric acid, but contains iron or manganese together with compounds of calcium or magnesium, it is saturated with chlorine, digested with carbonate of baryta, and the precipitate treated as directed in 12 or 13, for the separation of alumina from oxide of iron; or, as directed in 26, for the separation of manganese, the precipitate is then dissolved by hydrochloric acid, baryta separated as sulphate from the solution, and alumina separated from oxide of iron, as in 12 or 13; or,

36. It may be mixed with chloride of ammonium and an excess of sulphide of ammonium, the precipitate washed, dissolved by hydrochloric acid, manganese separated as directed in 26, and the precipitate treated as above directed.

37. When the solution contained phosphoric acid and compounds of iron or manganese, together with compounds of calcium or magnesium, and the phosphoric acid has been separated as basic perphosphate, the residue, *p.* 4 or *p.* 5, and the filtrate from the basic perphosphate are treated as

directed in 20, and the filtrate from the silica treated as directed in 15, 16, 17, 18, or 27.

38. When alumina is combined with phosphoric acid only, the residue, *p.* 2, is dissolved by hydrochloric acid, the solution evaporated to dryness, so as to separate silica, and alumina precipitated from the filtrate as above directed.

Calcium; estimated as carbonate, which is obtained by igniting the oxalate of lime, precipitated by oxalate of ammonia in the presence of free ammonia.

The solution is mixed with chloride of ammonium and ammonia, heated to the boiling-point, solution of oxalate of ammonia added until no further precipitate is produced, the liquid kept for twelve hours at a moderate temperature, the clear liquid then decanted off, the precipitate washed with hot water several times by decantation, then collected upon a filter, washed, dried, and ignited. The residue left after ignition is moistened with a few drops of carbonate of ammonia solution, dried and again slightly ignited before being weighed.

39. *Separation of calcium from potassium, sodium, or magnesium.*—When the solution does not contain compounds of any other metals besides calcium, than potassium, sodium, or magnesium, the precipitation by oxalate of ammonia may be effected at once, and without any special precautions.

40. When the solution does not contain phosphoric acid, but contains compounds of iron, manganese, or aluminum, lime is precipitated, as oxalate, from the filtrate from the sesquioxides or sulphides, as above directed (11, 23, 33, and 34).

41. When lime is combined with phosphoric acid only, it is estimated by weighing the precipitate, *p.* 1, of sulphate of lime (3).

Magnesium; estimated as pyrophosphate of magnesia ($2MgO,PO^5$) obtained by igniting the phosphate of magnesia and ammonia ($2MgO,NH^4O,PO^5$) which is precipitated from a part of the filtrate, *f.* 1.

The liquid is mixed with chloride of ammonium and ammonia, and phosphate of soda added in excess; it is then left for twenty-four hours at a moderate temperature, the clear liquid decanted off and filtered, the precipitate washed by decantation with water containing ammonia, transferred to a

crucible, dried, ignited, mixed with the ash of the filter, that has been separately incinerated on a piece of platinum foil, and weighed. The quantity, x, of magnesia is calculated from, q, that of pyrophosphate obtained, by the proportion:—

$$x : q = MgO : 2MgO,PO^5.$$

42. *Separation of magnesium from potassium or sodium.*—When the solution does not contain compounds of any other metals, and these are in the state of chlorides, it is concentrated by evaporation, mixed with oxide of mercury, sufficient to convert the whole of the chloride of magnesium into magnesia, and evaporated to perfect dryness, the residue is ignited until it ceases to give off vapour, digested with water, the magnesia collected upon a filter, washed, dried, ignited, and weighed; or,

43. The liquid is mixed with excess of baryta water, heated to the boiling-point, the precipitate separated by filtration, dissolved by hydrochloric acid, baryta separated as sulphate from the solution, and the magnesia precipitated as phosphate of magnesia and ammonia, as above directed.

44. When magnesia is combined with phosphoric acid alone, the filtrate, *f.* 2, is mixed with hydrochloric acid in slight excess, the precipitate separated by filtration and washed. The filtrate is mixed with chloride of ammonium and ammonia, the precipitate collected rapidly without access of air, washed, and the magnesia precipitated as phosphate of magnesia and ammonia, from the filtrate, as above directed.

45. When the solution contained lime with or without phosphoric acid and other bases, magnesia is precipitated, as phosphate of magnesia and ammonia, from the filtrate, from oxalate of lime (39).

Potassium; estimated, in a part of the filtrate, *f.* 1, as sulphate, chloride, or as double chloride of platinum and potassium.

Estimation as sulphate.—When the solution does not contain soda, it is evaporated to dryness, in a thin platinum capsule, the residue gradually heated to redness, and weighed.

When the solution contained an excess of sulphuric acid, the residue left after ignition contains bisulphate of potash, which must be converted into neutral sulphate; for this

purpose, when the crucible has cooled, a small piece of dry carbonate of ammonia is laid upon the saline mass, and heat applied very gradually until the crucible becomes red-hot: this operation is repeated until the weight of the mass does not decrease.

The quantity, x, of potassium, or, x', of potash, is calculated from, q, that of sulphate obtained, by the proportions:—

$$x : q = K : KO,SO^3; \text{ or, } x' : q = KO : KO,SO^3.$$

When the solution contains nitrate, chloride, or both salts, it must be heated with excess of sulphuric acid, in a porcelain basin, until nitric and hydrochloric acids are expelled, then transferred to the platinum capsule, evaporated to dryness, and the residue converted into neutral sulphate, as above directed.

Estimation as chloride.—When the solution contains only chloride of potassium it is evaporated to dryness, the residue very gradually heated to faint redness, and weighed.

Estimation as chloride of platinum and potassium.—The solution is evaporated to a small bulk, acidified with hydrochloric acid, mixed with alcoholic solution of bichloride of platinum, in excess, and with a considerable quantity of absolute alcohol; after standing twenty-four hours, the precipitate is collected upon a dry, weighed filter, washed with alcohol, dried, and weighed between watch glasses, ground at the edges and fastened by a light brass clamp, fig. 64.

Fig. 64.

The quantity of potassium or of potash is calculated from that of the double chloride obtained, by the proportions:—

$$x : q = K : PtCl^2,KCl; \text{ or, } x' : q = KO : PtCl^2,KCl.$$

When the solution contains ammoniacal salts, it must be evaporated to dryness, and the residue very gradually heated to redness, in a platinum capsule, until vapour is no longer given off. The ignited mass is dissolved in water, and the double chloride of platinum and potassium precipitated as above directed.

46. *Separation of potash from soda.*—The solution, evaporated to a small bulk, and free from ammoniacal salts, is mixed with considerable excess of bichloride of platinum and alcohol; after standing for twenty-four hours, the precipitate is collected upon a filter, washed with alcohol, dried,and weighed.

Sodium; estimated according to the state of combination in the solution, as sulphate, or chloride, in the same manner as potassium, and with the same precautions.

When sodium is associated with potassium, it is estimated in the alcoholic filtrate from the double chloride of platinum and potassium; this is evaporated to dryness in a platinum capsule, the residue ignited with a small quantity of oxalic acid, which facilitates the decomposition of the double chloride of platinum and sodium. The ignited mass is extracted with water, the platinum separated by filtration, the solution, which should be quite colourless, evaporated to dryness, and the residual chloride of sodium ignited and weighed.

The joint quantity of sodium and potassium should always be estimated, by weighing either the chlorides or sulphates, before the separation, so that a control of the separate estimation of each may be obtained, or, the quantity, x, of sodium may be estimated from the difference, s, between the weight, w, of the mixed salts, and the quantity, p, of the potassium salt, calculated from the result obtained by the direct estimation of potassium. Thus, supposing the salts to be chlorides:—

$$w\ (= \mathrm{NaCl} + \mathrm{KCl}) - p\ (= \mathrm{KCl}) = s\ (= \mathrm{NaCl}),$$

and

$$x : s = \mathrm{Na} : \mathrm{NaCl}.$$

This indirect method is frequently the most convenient that can be adopted for the estimation of both alkalies. In the first instance they must be obtained in a uniform and definite state of combination, as sulphates or chlorides, the entire quantity, w, of salt, ascertained by weighing, then dissolved in water and the quantity, N, of sulphuric acid, or of chlorine, estimated, in the usual manner, as sulphate of baryta (pp. 78 and 176) or chloride of silver (p. 180).

The quantities, p, s, of sulphates of potash and soda, or p', s', of chlorides of potassium and sodium are calculated from w and N, the quantity of sulphuric acid or chlorine in the mixture, by the equations:—

$$s = \frac{N - (w \,.\, 0{\cdot}45977}{0{\cdot}10861}, \text{ or } s' = \frac{N - (w \,.\, 0{\cdot}45977}{0{\cdot}10361}$$

and $p = w - s'$, or $p' = w - s'$.

In these equations the constants are the per-centage amount of sulphuric acid in sulphate of potash (45·977), and the difference between the per-centage amounts of sulphuric acid in the sulphates of soda and potash, 10·361.

In the analysis of soils, ashes, &c., the alkalies are estimated in a part of the filtrate, *f.* 1, which is mixed with excess of baryta water, warmed and filtered, the precipitate —containing the whole of the bases phosphoric and sulphuric acids—washed, excess of baryta separated, as sulphate, from the filtrate, *f.* 4, which is then evaporated to dryness, and the residue heated, so as to separate excess of sulphuric acid, and with carbonate of ammonia (p. 251) to convert bisulphates into neutral sulphate, the weight ascertained, and the amounts of potash and soda estimated as above directed; or,

When it is desired to estimate potash directly, the excess of baryta is separated from the filtrate, *f.* 4, by means of carbonate of ammonia, the liquid evaporated to dryness, the residue ignited, so as to separate ammoniacal salts, dissolved in water and potash precipitated as double chloride of platinum and potassium, as above directed.

Examination of the sulphuric acid solution.

This solution is treated in the same manner as the hydrochloric acid solution for the detection and estimation of the several constituents.

The insoluble residue which is left, may consist solely of silica, or of silica and undecomposed substance. To ascertain this, it is boiled with a dilute solution of potash or soda, which, in the former case, dissolves the whole.

Examination of the hydrofluoric solution.

The detection and estimation of the several constituents are effected in the same manner as in the case of the hydrochloric solution, except that the silica having been dissipated as fluoride of silicium, its amount is estimated from the difference between the quantity of substance operated upon and the sum of the quantities of the several constituents.

Examination of the solution obtained by fusion with alkali.

The whole of the liquid together with any undissolved silica is evaporated to dryness, and silica separated as directed at p. 239. The other operations are the same as in the examination of the hydrochloric solution, except that the alkalies must be estimated from the difference between the quantity of substance operated upon and the sum of the several constituents.

Reduction of results obtained in analysis.

It very frequently happens that the substances obtained in analysis are compounds or constituents of the substances sought, and in either case the quantities of the latter must be ascertained by calculation.

The following tables obviate the necessity of this calculation. Under the heads of the several elements are the names of the substances obtained in analysis, and the numbers in each column represent the quantities of the substances sought, that are equivalent to from 1 to 9 parts of the substances obtained. The quantities of substances sought, that are equivalent to any decimal multiple or fraction of 1, 2, 3, ... 9 parts of the substances obtained, are ascertained by making a corresponding change in the decimal point in these numbers. When the quantity of substance obtained is other than a decimal multiple or fraction of the numbers in the first column, the quantity of substance sought is found by adding together the quantities equivalent to each of the parts.

Thus, for instance, the quantities of aluminum equivalent to 1, 3, or 5 parts of alumina are 0·53307, 1·59922, 2·66537; the quantities equivalent to 30, 0·5 or 0·003 parts are 15·9922, 0·266537, 0·00159922, and the quantity equivalent to 3·215 parts alumina is 1·71382, for—

3·0	=	1·59922
0·2	=	0·10661
0·01	=	0·00533
0·005	=	0·00266
3·215	=	1·71382.

Element	ALUMINUM 13·7.	ANTIMONY 120.					
Substance obtained	Alumina.	Sulphide of antimony.		Antimoniate of oxide of antimony.			
Formula	Al^2O^3.	SbS^3.		Sb^2O^4.			
Substance sought	Aluminum.	Antimony.	Oxide of antimony.	Antimony.	Oxide of antimony.	Antimonic acid.	Sulphide of antimony.
	Al.	Sb.	SbO^3.	Sb.	SbO^3.	SbO^5.	SbS^3.
1.	0·53307	0·71428	0·85714	0·88235	1·05883	1·17647	1·23529
2.	1·06615	1·42857	1·71428	1·76470	2·11764	2·35294	2·47059
3.	1·59922	2·14285	2·57143	2·64706	3·17647	3·52941	3·70588
4.	2·13229	2·85714	3·42857	3·52941	4·23529	4·70588	4·94117
5.	2·66537	3·57142	4·28571	4·41176	5·29411	5·88235	6·17647
6.	3·19844	4·28571	5·14285	5·29413	6·35294	7·05882	7·41176
7.	3·73152	4·99999	6·00000	6·17647	7·41176	8·23529	8·64706
8.	4·26459	5·71428	6·85714	7·05882	8·47058	9·41176	9·88235
9.	4·79766	6·42856	7·71428	7·94117	9·52941	10·58823	11·11764

Element	ARSENIC 75.					
Substance obtained	Arsenous acid.	Sulphide of arsenic.	Arsenate of magnesia and ammonia.			
Formula	AsO^3.	AsS^3.	$\left.\begin{matrix}2MgO\\NH^4O\end{matrix}\right\}AsO^5,+HO.$			
Substance sought	Arsenic. As.	Arsenic. As.	Arsenic. As.	Arsenous acid. AsO^3.	Arsenic acid. AsO^5.	Sulphide of arsenic. AsS^3.
1.	0·75757	0·60975	0·39473	0·52105	0·60526	0·64737
2.	1·51515	1·21951	0·78947	1·04210	1·21052	1·29473
3.	2·27272	1·82927	1·18421	1·56315	1·81579	1·94210
4.	3·03030	2·43902	1·57891	2·08421	2·42105	2·58947
5.	3·78788	3·04878	1·97368	2·60526	3·02631	3·23684
6.	4·54545	3·65853	2·36848	3·12631	3·63158	3·88421
7.	5·30302	4·26829	2·76316	3·64736	4·23684	4·53158
8.	6·06060	4·87805	3·15789	4·16841	4·84210	5·17895
9.	6·81818	5·48780	3·55262	4·68947	5·44737	5·82631

Element	BARIUM 68·5.		BISMUTH 208.	BROMINE 80.	CALCIUM 20.			
Substance obtained	Sulphate of baryta.		Oxide of bismuth.	Bromide of silver.	Carbonate of lime.		Sulphate of lime.	
Formula	BaO,SO^3.		BiO^3.	AgBr.	CaO,CO^2.		CaO,SO^3.	
Substance sought	Barium. Ba.	Baryta. BaO.	Bismuth. Bi.	Bromine. Br.	Calcium. Ca.	Lime. CaO.	Calcium. Ca.	Lime. CaO.
1.	0·58798	0·65665	0·89655	0·42553	0·40000	0·56000	0·29412	0·41176
2.	1·17596	1·31330	1·79310	0·85106	0·80000	1·12000	0·58823	0·82353
3.	1·76395	1·96995	2·68965	1·27659	1·20000	1·68000	0·88235	1·23529
4.	2·35193	2·62661	3·58620	1·70213	1·60000	2·24000	1·17647	1·64706
5.	2·93991	3·28326	4·48276	2·12766	2·00000	2·80000	1·47059	2·05882
6.	3·52789	3·93991	5·37931	2·55319	2·40000	3·36000	1·76470	2·47059
7.	4·11588	4·59656	6·27586	2·97872	2·80000	3·92000	2·05882	2·88235
8.	4·70386	5·25322	7·17241	3·40425	3·20000	4·48000	2·35294	3·29412
9.	5·29184	5·90987	8·06896	3·82979	3·60000	5·04000	2·64716	3·70588

Element	CARBON 6.	CHLORINE 35·5.		CHROMIUM 27·7.			COPPER 31·7.
Substance obtained	Carbonic acid.	Chloride of silver.		Oxide of chromium.		Chromate of lead.	Oxide of copper.
Formula	CO^2.	AgCl.		Cr^2O^3.		PbO,CrO^3.	CuO.
Substance sought	Carbon.	Chlorine.	Hydrochloric acid.	Chromium.	Chromic acid.	Chromic acid.	Copper.
	C.	Cl.	HCl.	Cr.	CrO^3.	CrO^3.	Cu.
1.	0·27273	0·24738	0·25435	0·69773	1·30227	0·31640	0·79849
2.	0·54545	0·49477	0·50871	1·39546	2·60453	0·63280	1·59698
3.	0·81818	0·74216	0·76306	2·09320	3·90680	0·94920	2·39546
4.	1·09091	0·98954	1·01742	2·79093	5·20907	1·26560	3·19395
5.	1·36363	1·23693	1·27178	3·48866	6·51133	1·58201	3·99244
6.	1·63636	1·48432	1·52613	4·18639	7·81360	1·89841	4·79093
7.	1·90909	1·73170	1·78049	4·88412	9·11587	2·21481	5·58942
8.	2·18182	1·97909	2·03484	5·58185	10·41813	2·53121	6·38791
9.	2·45454	2·22648	2·28920	6·27959	11·72040	2·84761	7·18640

Element	HYDROGEN 1.	IODINE 127.		IRON 28.		LEAD 103·7.		
Substance obtained	Water.	Iodide of silver.	Iodide of palladium.	Oxide of iron.		Oxide of lead.	Sulphate of lead.	
Formula	HO.	AgI.	PdI.	Fe^2O^3.		PbO.	PbO,SO^3.	
Substance sought	Hydrogen. H.	Iodine. I.	Iodine. I.	Iron. Fe.	Protoxide of iron. FeO.	Lead. Pb.	Lead. Pb.	Oxide of lead. PbO.
1.	0·11111	0·54042	0·70438	0·70000	0·90000	0·92838	0·68358	0·73632
2.	0·22222	1·08085	1·40876	1·40000	1·80000	1·85676	1·36717	1·47264
3.	0·33333	1·62127	2·11314	2·10000	2·70000	2·78514	2·05076	2·20896
4.	0·44444	2·16170	2·81752	2·80000	3·60000	3·71352	2·73434	2·94528
5.	0·55555	2·70213	3·52190	3·50000	4·50000	4·64190	3·41793	3·68161
6.	0·66666	3·24255	4·22628	4·20000	5·40000	5·57028	4·10151	4·41793
7.	0·77778	3·78298	4·93067	4·90000	6·30000	6·49865	4·78510	5·15425
8.	0·88889	4·32340	5·63505	5·60000	7·20000	7·42703	5·46869	5·89057
9.	1·00000	4·86383	6·33943	6·30000	8·10000	8·35541	6·15227	6·62689

Element	MAGNESIUM 12.			MANGANESE 27·6.			
Substance obtained	Magnesia.	Sulphate of magnesia.		Protosesquioxide of manganese.			
Formula	MgO.	MgO, SO^3.		MnO + Mn^2O^3.			
Substance sought	Magnesium.	Magnesium.	Magnesia.	Manganese.	Oxide of manganese.	Sesquioxide of manganese.	Peroxide of manganese.
	Mg.	Mg.	MgO.	Mn.	MnO.	Mn^2O^3.	MnO^2.
1.	0·60000	0·20000	0·33333	0·72125	0·93031	1·03484	1·13937
2.	1·20000	0·40000	0·66666	1·44251	1·86063	2·06968	2·27874
3.	1·80000	0·60000	1·00000	2·16376	2·79094	3·10453	3·41812
4.	2·40000	0·80000	1·33333	2·88502	3·72125	4·13937	4·55749
5.	3·00000	1·00000	1·66666	3·60627	4·65157	5·17421	5·69686
6.	3·60000	1·20000	2·00000	4·32752	5·58188	6·20906	6·83623
7.	4·20000	1·40000	2·33333	5·04878	6·51219	7·24390	7·97561
8.	4·80000	1·60000	2·66666	5·77003	7·44251	8·27874	9·11498
9.	5·40000	1·80000	3·00000	6·49129	8·37282	9·31359	10·25435

Element	MERCURY 100.		NICKEL 29·6.	NITROGEN 14.					
Substance obtained	Mercury.		Oxide of nickel.	Chloride of platinum and ammonium.			Platinum.		
Formula	Hg.		NiO.	$PtCl^2, NH^4Cl$.			Pt.		
Substance sought	Sub-oxide.	Oxide of mercury.	Nickel.	Nitrogen.	Ammonia.	Oxide of ammonium.	Nitrogen.	Ammonia.	Oxide of ammonium.
	Hg^2O.	HgO.	Ni.	N.	NH^3.	NH^4O.	N.	NH^3.	NH^4O.
1.	1·04000	1·08000	0·78723	0·06272	0·07616	0·11649	0·14183	0·17224	0·26342
2.	2·08000	2·16000	1·57447	0·12545	0·15233	0·23297	0·28367	0·34448	0·52685
3.	3·12000	3·24000	2·36170	0·18817	0·22849	0·34946	0·42550	0·51672	0·79027
4.	4·16000	4·32000	3·14893	0·25089	0·30465	0·46595	0·56733	0·68895	1·05369
5.	5·20000	5·40000	3·93617	0·31362	0·38082	0·58243	0·70917	0·86119	1·31712
6.	6·24000	6·48000	4·72340	0·37634	0·45698	0·69892	0·85100	1·03343	1·58054
7.	7·28000	7·56000	5·51064	0·43907	0·53315	0·81541	0·99284	1·20567	1·84397
8.	8·32000	8·64000	6·29787	0·50179	0·60931	0·93189	1·13467	1·37791	2·10739
9.	9·36000	9·72000	7·08510	0·56451	0·68547	1·04838	1·27650	1·55015	2·37081

Element	PHOSPHORUS 31.			POTASSIUM 39.					
Substance obtained	Pyrophosphate of magnesia.		Phosphate of iron.	Sulphate of potash.		Chloride of potassium.		Chloride of platinum and potassium.	
Formula	$2MgO,PO^5$.		Fe^2O^3,PO^5.	KO,SO^3.		KCl.		$PtCl^2KCl$.	
Substance sought	Phosphorus. P.	Phosphoric acid. PO^5.	Phosphoric acid. PO^5.	Potassium. K.	Potash. KO.	Potassium. K.	Potash. KO.	Potassium. K.	Potash. KO.
1.	0·27928	0·63964	0·47020	0·44827	0·54023	0·52349	0·63087	0·15970	0·19246
2.	0·55856	1·27928	0·94040	0·89655	1·08046	1·04698	1·26174	0·31941	0·38493
3.	0·83784	1·91892	1·41059	1·34483	1·62069	1·57047	1·89261	0·47911	0·57739
4.	1·11711	2·55856	1·88079	1·79310	2·16092	2·09395	2·52349	0·63882	0·76986
5.	1·39639	3·19820	2·35099	2·24138	2·70115	2·61744	3·15436	0·79852	0·96232
6.	1·67567	3·83784	2·82119	2·68965	3·24138	3·14093	3·78523	0·96823	1·15479
7.	1·95495	4·47748	3·29139	3·13793	3·78161	3·66442	4·41610	1·11793	1·34725
8.	2·23423	5·11711	3·76159	3·58620	4·32184	4·18791	5·04697	1·27764	1·53972
9.	2·51351	5·75675	4·23179	4·03448	4·86207	4·71140	5·67785	1·43734	1·73218

Element	SILICON 14·2.	SILVER 108.	SODIUM 23.				STRONTIUM 43·8.	
Substance obtained	Silica.	Chloride of silver.	Sulphate of soda.		Chloride of sodium.		Sulphate of strontia.	
Formula	SiO^2 or SiO^3.	AgCl.	NaO, SO^3.		NaCl.		SrO, SO^3.	
Substance sought	Silicon.	Silver.	Sodium.	Soda.	Sodium.	Soda.	Strontium.	Strontia.
	Si.	Ag.	Na.	NaO.	Na.	NaO.	Sr.	SrO.
1.	0·47020	0·75261	0·32394	0·43662	0·39316	0·52991	0·47712	0·56427
2.	0·94040	1·50522	0·64789	0·87324	0·78632	1·05983	0·95425	1·12854
3.	1·41059	2·25784	0·97183	1·30986	1·17949	1·58974	1·43137	1·69281
4.	1·88079	3·01045	1·29577	1·74648	1·57265	2·11966	1·90849	2·25708
5.	2·35099	3·76306	1·61972	2·18310	1·96581	2·64957	2·38562	2·82135
6.	2·82119	4·51568	1·94366	2·61972	2·35897	3·17949	2·86274	3·38562
7.	3·29139	5·26829	2·26760	3·05634	2·75213	3·70940	3·33987	3·94989
8.	3·76159	6·02090	2·59155	3·49296	3·14530	4·23931	3·81699	4·51416
9.	4·23179	6·77352	2·91549	3·92958	3·53846	4·76923	4·29412	5·07843

Element	SULPHUR 16.				TIN 58.		ZINC 32·6.
Substance obtained	Sulphate of baryta.				Binoxide.		Oxide of zinc.
Formula	BaO,SO^3.				SnO^2.		ZnO.
Substance sought	Sulphur. S.	Sulphide of hydrogen. HS.	Sulphurous acid. SO^2.	Sulphuric acid. SO^3.	Tin. Sn.	Oxide of Tin. SnO.	Zinc. Zn.
1.	0·13734	0·14592	0·27468	0·34335	0·78378	0·89189	0·80295
2.	0·27468	0·29184	0·54935	0·68669	1·56757	1·78378	1·60591
3.	0·41202	0·43777	0·82403	1·03004	2·35135	2·67567	2·40886
4.	0·54935	0·58369	1·09871	1·37339	3·13513	3·56757	3·21182
5.	0·68669	0·72961	1·37339	1·71674	3·91892	4·45946	4·01478
6.	0·82403	0·87553	1·64807	2·06008	4·70270	5·35135	4·81773
7.	0·96137	1·02146	1·92274	2·40343	5·48648	6·24324	5·62069
8.	1·09871	1·16738	2·19742	2·74678	6·27027	7·13513	6·42364
9.	1·23605	1·31330	2·47213	3·09013	7·05405	8·02702	7·22660

CHAPTER X.

SYSTEMATIC QUALITATIVE ANALYSIS.

THE system of analysis laid down in this chapter is applicable to all inorganic substances and most compounds of organic acids that are of frequent occurrence in commerce.

The methods of testing for substances that are of rare occurrence are omitted, for the sake of rendering the system more especially applicable to ordinary technical practice.

The constituents that, in ordinary practice, are likely to be met with in substances whose chemical nature is sought to be ascertained, may be divided into three groups:—

I. *Basic or electro-positive substances.*

POTASSIUM, SODIUM, AMMONIUM, MAGNESIUM, CALCIUM, STRONTIUM, BARIUM, ALUMINUM, CHROMIUM, ZINC, CADMIUM, MANGANESE, IRON, COBALT, NICKEL, URANIUM, PLATINUM, GOLD, TIN, ANTIMONY, ARSENIC, BISMUTH, COPPER, LEAD, MERCURY, SILVER.

II. *Electro-negative substances.*

OXYGEN, CHLORINE, BROMINE, IODINE, CYANOGEN, FLUORINE, SULPHUR, PHOSPHORUS, CARBON.

III. *Acids.*

SULPHURIC, PHOSPHORIC, ARSENOUS, ARSENIC, CARBONIC, SILICIC, BORACIC, CHROMIC, CHLORIC, OXALIC, TARTARIC, CITRIC, MALIC, BENZOIC, SUCCINIC, ACETIC, or FORMIC ACIDS.

It is highly improbable that all, or any large majority of the members of these groups should be present in any substance to be analysed. It will, in most instances, consist of one or more of the first group combined either with one or more of the second group, or, in the state of oxide, with one or more of the third group. It may likewise be a

definite chemical compound, or a mixture of two or more such compounds.

The object of the qualitative analysis is therefore twofold: to ascertain, viz.—

1. what are the constituents—elementary or other—and
2. their state of combination or of mixture.

The external physical characters of a substance—colour, state of aggregation, density, taste, smell, &c.,—will frequently warrant some general inference as to its chemical nature, especially in the case of homogeneous substances, but such indications must not be estimated at more than their true value, which consists merely in serving, more or less, as a guide in the actual analysis.

The systematic course of qualitative analysis applies only to solutions, and the chemical nature of some members of the above groups precludes the possibility of their existing together in solution, so that, in addition to the general improbability of the presence of a great number of constituents, the presence of some will be a sufficient indication of the absence of others—thus, for instance, sulphuric acid cannot be present in a substance containing baryta, and soluble in water, nor can silver be present in a substance soluble in hydrochloric acid, gold in a solution containing proto-compounds of tin or iron, and the reverse.

For the analysis of metallic substances and some others, in which the number and kind of constituents are always very limited, the special methods, described at pp. 158-163, 170-175, and in Chapter IX., may be advantageously adopted.

Preliminary examination.

Besides the indications that may be furnished by the physical characters of a substance, there are several others which may be obtained by submitting it to two or three simple tests,—for instance, to ascertain its behaviour with solvents, and when heated, either alone, or together with other substances. This preliminary examination should never be omitted in the analysis of a substance of unknown chemical nature, because a knowledge of its results will

often considerably reduce the number of necessary experiments, and ensure a great saving of time.

When the substance to be examined is solid:—

I. *A portion is powdered, covered with water in a test-tube and boiled.*

When the substance appears little altered, a portion of the clear filtered liquid is evaporated to dryness upon platinum foil to ascertain whether any substance is dissolved.

Note.—In some instances a very small residue may be obtained in this way, arising either from the admixture of a minute quantity of soluble substance, or from the substance being very sparingly soluble. This may generally be disregarded, and the substance treated as if insoluble in water. Sulphate of lime, which requires 430 parts of water for solution, may in practice be taken as the limit of solubility in water.

a. *Substances that prove insoluble in water, or the portion that remains undissolved, must be heated with hydrochloric acid.*

Attention should be paid to any reaction that may take place when the acid is added.

1. **Effervescence** ..... { presence of **carbonates, sulphides, peroxides, cyanides.**

Test the gas with lime water, acetate of lead, iodide of potassium, and starch, and observe the smell.

2. **Complete solution** .... { absence of **silver, mercury** compounds corresponding to Hg_2O, and probably of **lead.**

b. *Substances that prove insoluble in water and hydrochloric acid are heated with nitric acid or nitrohydrochloric acid.*

c. *Substances that prove insoluble in water and acids are submitted to the preliminary tests described under* II. *and* III.

Such substances will most likely consist of one or other of the saline compounds indicated by **I** in the table of

relation to solvents. The method by which they are to be rendered soluble depends upon their composition.

When the substance does not contain metallic compounds that would be reduced and injure a platinum crucible, it may be melted with alkaline carbonate, the melted mass digested with water, and the residue boiled with nitric acid.

When the substance contains metals, it is best to boil it with caustic alkali, and then treat any residue that may remain, with nitric acid. The solutions thus obtained are examined as directed below.

When the substance to be examined consists of silicates, the method of analysis described at pp. 230, 235 are to be adopted.

When the substance is metallic, a special method of analysis (p. 158) is to be adopted.

When the substance is liquid, it is to be tested with litmus paper to ascertain whether it is acid or alkaline. Any peculiarities of colour, smell, or taste should be noticed.

A portion is evaporated to dryness in a platinum capsule.

Any residue that may be left is examined in the same way as a solid.

When it is desirable to obtain the liquid apart from any substance that may be held in solution, it should be distilled from a small flask.

When the liquid has an acid reaction and leaves a residue on evaporation, a portion should be mixed with a large quantity of water.

3. **It becomes opalescent,** the precipitate redissolving again on the addition of hydrochloric acid

{ presence of **antimony, bismuth,** or **tin.**

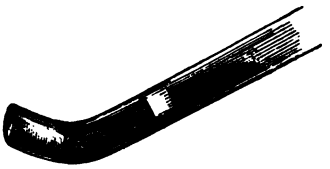
II. *A small portion of the substance is heated upon platinum foil.*

4. **It volatilizes,** either wholly or partially.

To obtain further indications of the nature of the vola-

tilizable substance, a portion is heated in a dry test-tube, or in a tube open at both ends (fig. 65).

Fig. 65.



a. Clear colourless drops collect at the upper part of the tube ..... **water.**

Note.—These drops should be tested with litmus, so as to ascertain whether the liquid is acid or alkaline.

b. A grey film is deposited, and when rubbed with a feather metallic globules appear ..... **mercury.**

c. A dark lustrous metallic film is deposited, and an odour resembling garlic evolved ..... **arsenic.**

Note.—When an open tube is used, the presence of arsenic is indicated by a white sublimate of arsenous acid, from which the metallic sublimate may be produced by heating it with black flux.

d. A yellow film is deposited and a sulphurous odour evolved ..... **sulphur.**

Note.—A yellow sublimate may in some instances consist of sulphide of arsenic.

e. A white sublimate is deposited } **antimony** or **arsenic.**

Note.—A white sublimate may also be produced in some instances by mercurial compounds, chloride of lead, oxide of bismuth, sulphides of lead or tin, and some ammoniacal salts.

When a substance contains sulphur or arsenic, it is advisable

to separate them before applying the tests described in 7; this is best effected by roasting the powdered substance in an open tube or in the outer blowpipe flame on charcoal. Sometimes the separation of sulphur or arsenic is difficult, and in such cases the substance should be frequently powdered during the roasting. Alternate oxidation and reduction by means of charcoal powder, is sometimes advantageous in facilitating the separation of sulphur and arsenic.

5. **It melts readily,** either with or without expulsion of vapour ..... { presence of saline compounds of the **alkaline** or **earthy metals.**

Note.—As a test for nitrates or chlorates, a piece of paper is dipped into the melted substance. The presence of these salts is indicated by deflagration.

6. **It chars,** or blackens and becomes lighter coloured again when strongly ignited ..... { presence of **fixed organic substance.**

Note.—The coal should be strongly heated so as to burn off the carbon; when fixed mineral substance is present, it remains as ash, and may be subjected to the further preliminary tests.

When a substance contains organic admixtures, it is advisable to destroy them by ignition, because in many cases the normal reactions of inorganic substances are prevented or modified by the presence of organic substances.

7. **It acquires a darker colour,** that is permanent after strong ignition ..... { presence of compounds of the **heavy metals.**

To obtain further indications of the nature of the substance, small portions of the residue—that has been well roasted when the substance contains sulphur or arsenic—are melted with borax or microcosmic salt, both in the outer and inner blowpipe-flame. The colour presented by the beads thus obtained is often indicative of the metals present, as shown in the following table.

In order to test substances by fusion with borax or microcosmic salt, a piece of thin platinum wire, bent at the end,

as shown in fig. 66, is heated to redness and dipped into the salt; the portion that adheres is melted in the outer blow-

Fig. 66.

pipe-flame, and this operation repeated until the bead is large enough to fill the loop of wire. It should be perfectly colourless when cold.

A very small fragment of the substance to be examined is attached to the bead by moistening its surface, and it is then brought into the outer blowpipe-flame and melted. Any reaction that may take place during the fusion is noted, as well as the colour of the bead while hot and when cold.

The colour of a bead is generally ascertained best by placing it in front of a piece of white paper. When the bead contains a substance that does not dissolve, it should be examined by means of a magnifying glass.

The bead is then to be melted in the reducing flame, either upon the platinum wire or on charcoal, and the same points noted.

Note.—When the substance is suspected to contain zinc, nickel, cadmium, lead, bismuth, copper, silver, or antimony, the reduced metal would alloy with the platinum, and to avoid this, the bead must be detached from it and melted upon charcoal in the reducing flame.

The action of borax in testing substances consists in the production of compounds which, whether containing excess of base or acid, are very fusible. The action of microcosmic salt consists chiefly in the solution of substances by the phosphoric acid it contains, and the formation of fusible double salts.

The colour of the bead produced with borax or microcosmic salt depends very often upon the proportion of the metallic oxide to which the colour is due. In all instances the experiment should be made at first with a very small quantity of substance, and, if desirable, repeated with a larger quantity, so as to produce a glass saturated with the oxide.

Oxidizing flame.		*Reducing flame.*		
Borax.	**Microcosmic salt.**	**Borax.**	**Microcosmic salt.**	
Intense violet while hot. Becomes red when cold.	Violet bead.	Colourless bead.	Colourless bead.	**Oxide of manganese.**
Yellow while hot. Colourless when cold.	Yellowish-red while hot. Yellow when cold.	Green bead.	Red bead.	**Oxide of iron.**
Blue.	Blue.	Blue.	Blue.	**Oxide of cobalt.**
Violet while hot. Red-brown when cold.	Reddish bead while hot. Yellow when cold.	Grey bead, turbid. Becomes colourless with a nucleus of metal.	Reddish bead while hot. Yellow when cold.	**Oxide of nickel.**
Colourless clear bead. Becomes opaque.	The same as with borax.	Opaque and turbid at first, afterwards clear.	The same as with borax.	**Oxide of zinc.**
Yellow clear bead while hot. Nearly colourless when cold. Becomes opaque.	The same as with borax.	Oxide reduced on charcoal. Yellow incrustation.	The same as with borax, but less marked.	**Oxide of cadmium.**
Yellow while hot. Colourless when cold.	The same as with borax.	Becomes turbid on charcoal, and finally clear.	Becomes grey on charcoal.	**Oxide of lead.**

clear bead. Becomes opaque when reheated.	clear bead.	partial reduction on charcoal.	charcoal or platinum wire.	**Oxide of tin.**
Yellow. Becomes opaque when cold.	Colourless clear bead.	Becomes grey and turbid; finally clear.	Remains colourless while hot on charcoal.	**Oxide of bismuth.**
Yellow while hot. Colourless when cold.	Yellow. Becomes greenish when cold.	Becomes green.	Becomes green.	**Oxide of uranium.**
Green while hot. Blue when cold.	The same as with borax.	Colourless while hot. Red when cold.	The same as with borax.	**Oxide of copper.**
Opaque bead.		Opaline bead.		**Oxide of silver.**
Yellowish while hot. Colourless when cold.	The same as with borax.	Grey opaque bead.	The same as with borax.	**Oxide of antimony.**
Yellow while hot. Greenish when cold.	Reddish while hot. Green when cold.	Green.	Reddish while hot. Green when cold.	**Oxide of chromium.**

8. **No apparent alteration,** either permanent or temporary; absence of **organic substance.**

When the substance leaves an infusible white residue, it should be moistened with cobalt solution and strongly heated upon charcoal by means of the oxidizing blowpipe-flame. Colour is sometimes produced in this way, that indicates the presence of some substances.

a. Rose colour	**magnesia.**
b. Blue	**alumina.**
c. Yellowish green.....	**oxide of zinc.**
d. Blueish green.....	**oxide of tin.**
e. Olive green.....	**oxide of antimony.**

III. *The substance is mixed with carbonate of soda and heated upon charcoal, in the reducing blowpipe-flame.*

9. **It melts,** and is entirely absorbed by the charcoal
{ presence of **alkaline salts**; absence of compounds of **other bases.**

10. **Metallic globules appear**
{ presence of **gold, silver, tin,** or **copper.**

11. **An incrustation is produced** on the charcoal, besides or without metallic globules
{ presence of **bismuth, lead, antimony, cadmium,** or **zinc.**

The colour of the incrustation may sometimes furnish a clue to the substances present.

a. A yellow incrustation, becoming white when cold	indicates **zinc.**
b. A yellow or brown incrustation	indicates **bismuth, lead,** or **cadmium.**
c. A white incrustation..	indicates **antimony.**

d. soft globules, easily flattened } indicate **lead.**

Note.—The incrustation produced by lead is very like that produced by bismuth, but the globules of bismuth are brittle.

e. brittle globules ..... { indicate either **bismuth** or **antimony.**

Note.—The globules of antimony burn after the blast has ceased, giving off vapour, and generally become enveloped with crystals.

12. **It is dissolved with effervescence** by the melted soda { presence of **silica.**

IV. *A portion of the substance is covered with concentrated sulphuric acid in a test-tube, and heated.*

13. **Effervescence** { indicates the presence of **carbonates, sulphides, sulphites,** or **cyanides.**

Observe the smell of the gas, and test it with lime water, acetate of lead, &c.

14. **Acid vapour is evolved** { presence of **chlorides, bromides, iodides,** or **nitrates.**

When the vapour is brown, test with starch solution;

a. A blue coloration .... indicates **iodides.**
b. An orange coloration.. indicates **bromides.**
c. No coloration..... indicates **nitrates.**

15. **The liquid becomes dark coloured** { presence of **organic substances.**

After the application of these preliminary tests, some general notion may be formed as to the chemical nature of the substance under examination and its probable constituents.

When the substance is solid, a quantity amounting to 1 or 2 grammes is then to be dissolved by such means as may have been found most suitable for the purpose, and the clear solution submitted to the operations described below.

Any residue that cannot be dissolved is separated by filtration and examined separately.

ACTUAL ANALYSIS.

It has already been remarked, that in qualitative analysis, the application of special tests must be preceded by operations whose object is to effect the separation of the elementary constituents of substances in groups, the members of each of which present some reaction that is characteristic and distinctive as regards other groups.

The analytical groups comprising the metallic elements are five in number, and two of them are subdivided into secondary groups;—the reagents, by means of which the constituents of a substance are separated in groups, are

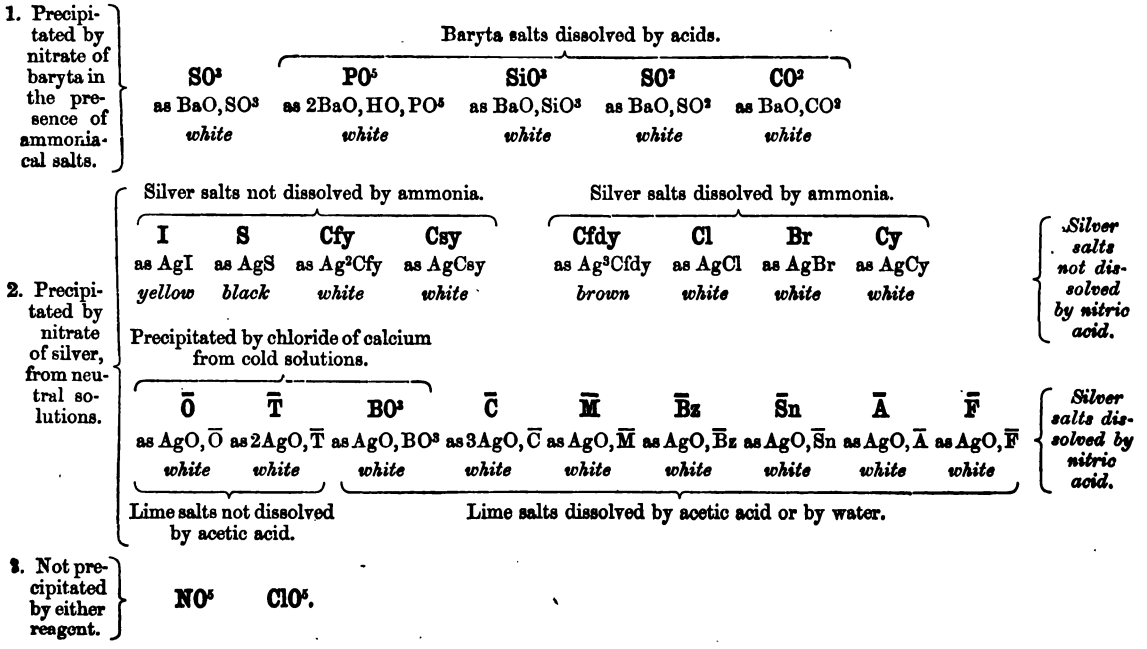
Hydrochloric acid,
Sulphuretted hydrogen,
Sulphide of ammonium, and
Carbonate of ammonia.

The analytical groups comprising the acids and electronegative elements are three in number, and one of them is subdivided into two secondary groups, each of which is again subdivided into two others;—the reagents, by means of which these constituents of a substance are separated in groups, are

Nitrate or *acetate of baryta*,
Nitrate of silver, and
Chloride of calcium.

The examination for acids is generally made to follow that for bases.

1. Precipitated by hydrochloric acid.	**Ag** as $AgCl$ *white*	**Hg** as Hg^2Cl *white*	**Pb** as $PbCl$ *white*					
2. Precipitated by sulph. hydrogen, from acid solutions.	**Hg** as HgS *black*	**Pb** as PbS *black*	**Bi** as BiS^3 *black*	**Cd** as CdS *yellow*	**Cu** as CuS *black*			*a. insoluble in sulphide of ammonium.*
	Au as AuS^3 *black*	**Pt** as PtS^2 *black*	**Sn** as SnS *black* or SnS^2 *yellow*	**Sb** as SbS^3 or SbS^5 *orange*	**As** as AsS^3 or AsS^5 *yellow*			*β. soluble in sulphide of ammonium.*
3. Precipitated by sulphide of ammonium, from alkaline solutions.	**Ni** as NiS *black*	**Co** as CoS *black*	**Mn** as MnS *flesh-coloured*	**Ur** as UrS *brown*	**Fe** as FeS *black*	**Cr** as Cr^2O^3 *blue-green*		*a.*
	Al as Al^2O^3 *white*	**Zn** as ZnS *white*						*β. oxides soluble in potash.*
4. Precipitated by carbonate of ammonia, from neutral solutions.	**Ba** as BaO, CO^2 *white*	**Sr** as SrO, CO^2 *white*	**Ca** as CaO, CO^2. *white*					
5. Remain in solution.	**Mg**	**K**	**Na.**					


1. Precipitated by nitrate of baryta in the presence of ammoniacal salts.
Baryta salts dissolved by acids.
SO³ as BaO,SO³ white
PO⁵ as 2BaO,HO,PO⁵ white
SiO³ as BaO,SiO³ white
SO² as BaO,SO² white
CO² as BaO,CO² white
2. Precipitated by nitrate of silver, from neutral solutions.
Silver salts not dissolved by ammonia.
Silver salts dissolved by ammonia.
I as AgI yellow
S as AgS black
Cfy as Ag²Cfy white
Csy as AgCsy white
Cfdy as Ag³Cfdy brown
Cl as AgCl white
Br as AgBr white
Cy as AgCy white
Silver salts not dissolved by nitric acid.
Precipitated by chloride of calcium from cold solutions.
Ō as AgO,Ō white
T̄ as 2AgO,T̄ white
BO³ as AgO,BO³ white
C̄ as 3AgO,C̄ white
M̄ as AgO,M̄ white
B̄z as AgO,B̄z white
S̄n as AgO,S̄n white
Ā as AgO,Ā white
F̄ as AgO,F̄ white
Silver salts dissolved by nitric acid.
Lime salts not dissolved by acetic acid.
Lime salts dissolved by acetic acid or by water.
3. Not precipitated by either reagent.
NO⁵
ClO⁵.

In applying the several tests it is advisable to operate upon a small portion of the filtrates, and, if a negative indication is obtained, to pass on to the next test, or if a positive indication is obtained, to treat the whole of the liquid in the same way.

Examination for basic or electro-positive constituents.

A. The solution is mixed with hydrochloric acid until it has a decided acid reaction.

1. *No precipitate* ..... { absence of **mercury, silver,** and probably of **lead.**

2. *White precipitate, insoluble in excess of the reagent*..... { may contain one or more metals belonging to the first group.

$AgCl$, Hg^2Cl, $PbCl$.

Note.—When the solution has an alkaline reaction, there may be an evolution of gas on the first addition of hydrochloric acid; evolution of carbonic acid, sulphuretted hydrogen, or hydrocyanic acid would indicate the presence of an alkaline carbonate, sulphide, or cyanide; a precipitate may also be produced, consisting of alumina, oxide of zinc, metallic sulphides, or cyanides, or some other substance soluble in alkali. When the solution has an acid reaction, a precipitate may be produced, consisting of antimonic acid, basic chlorides of antimony, or bismuth; but all these precipitates are readily distinguishable from the chlorides of silver, mercury, and lead, in being redissolved by an excess of hydrochloric acid. When the solution is neutral or alkaline, a precipitate may sometimes be produced by hydrochloric acid, consisting of benzoic or boracic acid, both of which redissolve when the liquid is warmed or diluted.

The precipitate, **P. 1,** is collected upon a filter, twice washed with water, the filtrate, **F. 1,** set aside for treatment with sulphuretted hydrogen.

The chloride precipitate, **P. 1,** is boiled with water in a small flask.

Complete solution ..... { absence of **mercury** and **silver.**

Insoluble residue..... { may contain **mercury** and **silver.**

The residue, **P. 2,** is separated by filtration while the liquid is hot, washed with boiling water, and the filtrate, **F. 2,** set aside for testing with sulphuric acid.

The residue, **P. 3**, is covered with solution of ammonia.

Complete solution	absence of **mercury.**
It becomes black ..... Hg^2O.	presence of **mercury.**

The filtrate **F. 3**, from the oxide of mercury or the ammoniacal liquid, is neutralized with nitric acid.

No precipitate.....	absence of **silver.**
White precipitate ..... AgCl.	presence of **silver.**

The filtrate, **F. 2**, is mixed with sulphuric acid.

No precipitate.....	probable absence of **lead.**
White precipitate, PbO,SO^3.	presence of **lead.**

Note.—A white precipitate with sulphuric acid indicates with certainty the presence of lead, but, when no precipitate is produced with sulphuric acid, the absence of lead must not be regarded as certain, because lead is not separated from dilute solutions by hydrochloric acid, and in such cases must be sought in the filtrate, **F. 1.**

B. The acid filtrate, **F. 1**, or the solution that has been mixed with excess of hydrochloric acid, is saturated with sulphuretted hydrogen and warmed for ten minutes.

No precipitate.....	absence of all elements belonging to the second group.
Coloured precipitate	may contain one or more metals belonging to the second group.

HgS, PbS, BiS^3, CuS, CdS,
AuS, PtS, SnS, SnS^2, AsS^3, AsS^5, SbS^3, SbS^5.

Note.—When the solution contains arsenic acid (AsO^5), it is advisable to saturate it with sulphurous acid and boil, before treatment with sulphuretted hydrogen. By this means arsenic acid is converted into arsenous acid (AsO^3), which is converted into sulphide much more readily than arsenic acid. A white precipitate, consisting of sulphate of lead, strontia, or baryta, may be produced by the sulphurous acid; this should be separated and warmed with hydrochloric acid, and the liquid mixed with that from which the precipitate was separated.

After perfect precipitation the sulphide precipitate, **P. 5,** is collected on a filter, washed with sulphuretted hydrogen water, and the filtrate, **F. 4,** set aside for treatment with sulphide of ammonium.

Note.—The colour of the precipitate produced by sulphuretted hydrogen will sometimes afford an indication of its probable nature. When the solution contains persalts of iron, chromic, chloric, bromic, iodic, or sulphurous acids, a precipitate of sulphur is formed with sulphuretted hydrogen, even in the absence of metals of the second group, but it is white, and therefore not likely to be mistaken for the positive indication, except in the case of solutions containing chromic acid, when the white precipitate may appear for a time green.

β. The sulphide precipitate, **P. 5,** is thoroughly washed, and digested in a flask with sulphide of ammonium.

Complete solution ..... { absence of metals belonging to the first division of the second group.

Coloured residue ..... { may contain one or more metals belonging to the first division of the second group.

HgS, PbS, BiS^3, CuS, CdS.

The insoluble residue, **P. 6,** is separated by filtration, and the filtrate, **F. 5,** set aside for subsequent examination.

The insoluble residue, **P. 6,** is thoroughly washed, and boiled with nitric acid in a flask.

Complete solution ..... absence of **mercury.**

Black residue ..... HgS. } presence of **mercury.**

Note.—When the solution contains tin and cadmium, this residue may contain a small quantity of the sulphides of both metals.

The residue, **P. 7,** is collected upon a filter, washed, and the filtrate, **F. 6,** set aside for treatment with sulphuric acid.

The residue, **P. 4,** is dissolved by nitro-hydrochloric acid, the solution mixed with excess of ammonia, and boiled with pieces of copper wire, upon which the mercury is precipitated, and is afterwards tested for by subliming in a dry test-tube.

The filtrate, **F. 6**, is mixed with dilute sulphuric acid, heated, and left for half an hour.

No precipitate.....	absence of **lead.**
White precipitate ..... PbO,SO^3.	presence of **lead.**

The precipitate, **P. 8**, is separated by filtration, dried, and tested with soda before the blowpipe to confirm the indication of lead.

The filtrate, **F. 7**, is mixed with excess of ammonia.

No precipitate.....	absence of **bismuth.**
White precipitate ..... BiO^3.	presence of **bismuth.**

The precipitate, **P. 9**, is separated by filtration, washed, pressed between paper, dissolved by hydrochloric acid, the solution evaporated to a small bulk, and mixed with a large quantity of water; a white precipitate remaining suspended in the liquid confirms the indication of bismuth.

The filtrate, **F. 8**, from the oxide of bismuth, or the solution that has been tested with ammonia, is mixed with excess of sulphuretted hydrogen, and the precipitate mixed with cyanide of potassium solution.

Complete solution	absence of **cadmium.**
Yellow residue ..... CdS.	presence of **cadmium.**

The precipitate, **P. 10**, is separated by filtration, washed, and the filtrate, **F. 9**, mixed with hydrochloric acid.

No precipitate.....	absence of **copper.**
Black precipitate ..... CuS.	presence of **copper.**

Note.—The presence of copper would generally be indicated by the colour of the filtrate, **F. 8**, or of the ammoniacal liquid that has been tested for bismuth; in the absence of copper, the precipitate, **P. 10**, would be of a pure yellow colour. Very minute quantities of copper may be tested for, by means of ferrocyanide of potassium, in a portion of the ammoniacal liquid, evaporated and mixed with acetic acid.

The filtrate, **F. 5,** is diluted with four times its volume of water, and mixed with dilute hydrochloric acid.

No precipitate..... { absence of metals belonging to the second division of the second group.

Coloured precipitate .... { may contain one or more metals belonging to the second division of the second group.

SnS^2, SbS^5, AsS^5.

The sulphide precipitate, **P. 12,** is separated by filtration, washed, dried, mixed with equal weights of dry carbonate of soda and nitrate of soda, and the mixture projected in small portions into twice the weight of nitrate of soda melted in a porcelain crucible. The cold melted mass is then digested with cold water.

Complete solution ..... { absence of **tin, antimony, gold,** and **platinum.**

Insoluble residue..... { may contain **tin, antimony, gold,** or **platinum.**

The residue, **P. 13,** is collected upon a filter, washed with alcohol (0·92 sp. gr.), and the filtrate, **F. 10,** set aside, apart from the washings, for subsequent examination.

The residue, **P. 13,** is boiled with caustic soda, the liquid mixed with an equal volume of alcohol, and left for half an hour.

Complete solution ..... { absence of **gold, antimony,** and **platinum.**

Insoluble residue..... { may contain **antimony, gold,** or **platinum.**

The residue, **P. 14,** is collected upon a filter, washed with alcohol, and the filtrate, **F. 11,** set aside for subsequent examination.

The residue, **P. 14,** is boiled with hydrochloric acid, and some tartaric acid added.

Complete solution .... absence of **gold** and **platinum.**

Insoluble residue..... may contain **gold** or **platinum.**

The residue, **P. 15,** is collected upon a filter, washed, and the filtrate, **F. 12,** set aside for subsequent examination.

The residue, **P. 15**, is dissolved by nitro-hydrochloric acid, the solution evaporated to dryness, the residue dissolved in a small quantity of water, and the solution mixed with protochloride of iron.

No precipitate.....	absence of **gold.**
Reddish-yellow precipitate Au.	presence of **gold.**

Note.—When the quantity of gold is very minute, the precipitate is not perceptible for some hours, but the liquid acquires a blue tinge when mixed with the protochloride of iron.

The precipitate, **P. 16**, is separated by filtration, the filtrate, **F. 13**, evaporated to dryness with chloride of ammonium, and the residue covered with alcohol.

Complete solution	absence of **platinum.**
Yellow residue $PtCl^2, NH^4Cl$.	presence of **platinum.**

The filtrate, **F. 10**, is mixed with a very slight excess of nitric acid and with nitrate of silver solution, and the liquid then covered with a layer of very dilute ammonia.

No precipitate.....	probable absence of **arsenic.**
Red precipitate $3AgO, AsO^5$.	presence of **arsenic.**

Note.—In order to obtain a more positive indication of the presence of arsenic, especially when the amount is very small, a portion of the filtrate, **F. 10**, is evaporated to dryness, the residue heated with a mixture of dry carbonate of soda and cyanide of potassium in a small bulbed tube. By this means the arsenic is reduced and sublimes as a dark metallic film.

The filtrate, **F. 11**, is mixed with hydrochloric acid in slight excess, evaporated to remove alcohol, then saturated with sulphuretted hydrogen, and heated.

No precipitate.....	absence of **tin.**
Yellow precipitate SnS^2.	presence of **tin.**

The precipitate, **P. 19**, is washed, dried, and melted with cyanide of potassium and alkaline carbonate by the reducing-

flame of the blowpipe, in order to confirm the indication of tin.

The filtrate, **F. 12**, is saturated with sulphuretted hydrogen, and heated.

No precipitate.....	absence of **antimony.**
Orange precipitate ..... Sb_2S^5.	presence of **antimony.**

When neither gold nor platinum is present, the following method may be adopted in testing for tin, antimony, and arsenic.

The precipitate, **P. 12**, is digested for half an hour with solution of sesquicarbonate of ammonia, at a moderate temperature, and frequently shaken meanwhile.

Complete solution	absence of **antimony** and probably of **tin.**
Insoluble residue.....	may contain **tin** or **antimony.**

Note.—In the presence of sulphides of arsenic, bisulphide of tin is dissolved by sesquicarbonate of ammonia solution; consequently when arsenic is present, complete solution does not positively indicate the absence of tin; and for this reason, the sulphide of arsenic, **P. 18^a**, must always be tested for tin as directed below.

The residue, **P. 13^a**, is separated by filtration, and washed with sesquicarbonate of ammonia solution.

The filtrate, **F. 10^a**, is mixed with excess of hydrochloric acid and sulphuretted hydrogen.

No precipitate.....	absence of **arsenic.**
Yellow precipitate ..... As_2S^5.	presence of **arsenic.**

The precipitate, **P. 18^a**, is collected upon a filter and washed; a portion is dissolved by ammonia, the solution evaporated to dryness, and the residue tested to confirm the indication of arsenic.

Another portion of this precipitate is melted with nitrate of potash, the mass digested with boiling water, the solution mixed with nitric acid, and boiled, in order to ascertain whether tin is present.

The residue, **P. 13^c**, is dissolved by nitro-hydrochloric acid, the solution mixed with excess of sesquicarbonate of ammonia solution, and boiled.

Complete solution ..... absence of **tin.**

Insoluble residue..... probable presence of **tin.**

The residue, **P. 19^c**, is separated by filtration, washed with sesquicarbonate of ammonia solution, and the filtrate, **F. 12^c**, set aside for subsequent examination.

The residue, **P. 19^c**, is dried, melted with cyanide of potassium; the reduced metal dissolved by hydrochloric acid, and the solution tested with chloride of mercury to confirm the indication of tin.

Note.—This confirmatory test is necessary, because the residue, **P. 19^c**, insoluble in sesquicarbonate of ammonia solution, may, in some cases, consist wholly or partially of oxide of antimony.

The filtrate, **F. 12^c**, is mixed with hydrochloric acid and excess of sulphuretted hydrogen.

No precipitate..... { probable absence of **antimony.**

Orange-coloured precipitate SbS^5. } presence of **antimony.**

C. The filtrate, **F. 4**, or the solution that has been tested with sulphuretted hydrogen, is mixed with excess of ammonia, and then with sulphide of ammonium.

No precipitate..... { absence of metals belonging to the third group.

Precipitate ..... { may contain one or more metals of the third group.

FeS; Cr^2O^3; UrS; MnS; NiS; CoS; Al^2O^3; ZnS,

Note.—The colour of the precipitate produced by sulphide of ammonium will sometimes afford an indication of its probable nature.

When the substance under examination is insoluble in water, the precipitate produced by sulphide of ammonium may contain phosphates or oxalates of baryta, strontja, lime, or magnesia, which must be specially tested for, as directed below.

The precipitate, **P. 21**, is separated by filtration, washed with water containing some sulphide of ammonium, and the filtrate, **F. 14**, set aside for treatment with carbonate of ammonia.

The precipitate, **P. 21,** is covered with dilute hydrochloric acid, upon the filter.

Complete solution ..... { probable absence of **cobalt** and **nickel.**

Black residue ..... CoS and NiS. } may contain **cobalt** and **nickel.**

The black residue, **P. 22,** is washed, and tested for cobalt and nickel before the blowpipe.

Note.—The sulphide of nickel (NiS) and the sulphide of cobalt (CoS) are not quite insoluble in dilute hydrochloric acid; consequently the absence of a residue, **P. 22,** does not positively indicate the absence of nickel and cobalt, and even when a residue remains, these metals must be sought for subsequently.

The solution, **F. 15,** is boiled with a few drops of nitric acid, so as to remove sulphuretted hydrogen and convert iron into the state of combination corresponding to Fe^2O^3, diluted with water, mixed with a considerable excess of caustic potash, and boiled.

Complete solution ..... { absence of all metals belonging to the first division of the third group.

Insoluble residue..... { may contain one or more metals belonging to the first division of the third group, and when the substance analysed is insoluble in water, the salts enumerated above,

Fe^2O^3; Ur^2O^3; Cr^2O^3; MnO; NiO; CoO.

The residue, **P. 23,** is separated by filtration, washed, and the filtrate, **F. 16,** set aside.

A portion of the residue, **P. 23,** is dissolved off the filter by warm hydrochloric acid; the solution mixed with some chloride of ammonium and an excess of ammonia.

Precipitate first formed redissolves completely.. { absence of **iron** and **uranium,** probable absence of **chromium.**

Insoluble residue..... { may contain **iron, uranium,** and **chromium.**

Note.—When the substance under examination is insoluble in water, the

residue, **P. 23**, may contain phosphates of magnesia, lime, strontia, or baryta, and in such instances the phosphoric acid may be separated, as directed at p. 242, from the solution of the residue, **P. 23.**

The precipitate, **P. 24**, is rapidly collected upon a filter, washed without access of air, and the filtrate, **F. 17**, set aside.

The precipitate, **P. 24**, is dissolved by hydrochloric acid, the solution neutralized with ammonia and mixed with carbonate of ammonia solution.

Precipitate first formed redissolves completely	absence of **iron** and probable absence of **chromium.**
Insoluble residue.....	may contain **iron** and **chromium.**

The precipitate, **P. 25**, is washed, and the filtrate, **F. 18**, set aside for subsequent examination.

The precipitate, **P. 25**, is dried, melted with 3 times its weight of nitrate of potash and its own weight of carbonate of soda in a porcelain crucible, and the mass digested with warm water.

Complete solution	absence of **i** .
Insoluble residue ..... Fe^2O^3.	presence of **iron.**

The filtrate, **F. 19**, is mixed with acetate of lead solution.

No precipitate.....	probable absence of **chromium.**
Yellow precipitate ..... PbO, CrO^3.	presence of **chromium.**

The filtrate, **F. 18**, or the solution that has been tested with carbonate of ammonia, is evaporated to a small bulk, neutralized with hydrochloric acid, and mixed with ammonia.

No precipitate.....	absence of **uranium.**
Yellow precipitate ..... $Ur^2O^3NH^3$.	presence of **uranium.**

The yellow precipitate, **P. 28**, is tested before the blowpipe to confirm the indication of uranium.

The filtrate, **F. 17,** is evaporated to dryness, with excess of hydrochloric acid, and nitric acid added meanwhile, so as to remove ammoniacal salts, the residue dissolved in water, the solution mixed with excess of carbonate of ammonia solution, and warmed.

No precipitate..... absence of **manganese.**

Flesh-coloured precipitate MnO, CO^2. } presence of **manganese.**

The filtrate, **F. 20,** from the carbonate of manganese is mixed with hydrochloric acid, then with cyanide of potassium solution, until the precipitate that may be produced is redissolved, the solution boiled, and hydrochloric acid added occasionally until hydrocyanic acid is no longer evolved; the solution then mixed with excess of potash, and again boiled for a few minutes.

No precipitate..... absence of **nickel.**

Pale-green precipitate.... NiO, HO. } presence of **nickel.**

The filtrate, **F. 21,** is mixed with nitric acid in excess, evaporated to dryness, the residue melted in a porcelain crucible, and the mass digested with water.

Complete solution ..... absence of **cobalt.**

Black residue..... CoO. } presence of **cobalt.**

Note.—The black residue, **P. 22,** left undissolved by hydrochloric acid, may be dissolved by nitro-hydrochloric acid, and the solution tested in the same manner for nickel and cobalt.

The filtrate, **F. 16,** is neutralized with hydrochloric acid, mixed with a very slight excess of ammonia, and heated.

No precipitate..... absence of **aluminum.**

White precipitate ..... Al^2O^3. } presence of **aluminum.**

Note.—The alumina precipitate may contain phosphoric acid, to detect which it is dissolved by hydrochloric acid, and tested for with molybdate of ammonia.

The precipitate, **P. 32**, is separated by filtration, and the filtrate, **F. 22**, mixed with sulphide of ammonium.

No precipitate..... absence of **zinc.**

White precipitate ..... } presence of **zinc.**
ZnS.

The filtrate, **F. 14**, or the solution that has been tested with sulphide of ammonium, is mixed with carbonate of ammonia solution.

No precipitate..... { absence of all the members of the fourth group.

White precipitate ..... { may contain one or more members of the fourth group.

BaO, CO^2; SrO, CO^2; CaO, CO^2.

The precipitate, **P. 34**, is collected upon a filter, washed, and the filtrate, **F. 23**, set aside for subsequent examination.

A portion of the precipitate, **P. 34**, is dissolved by hydrochloric acid, the solution mixed with hydrofluosilicic acid in excess, evaporated to a small bulk, and the residue mixed with alcohol.

No precipitate..... absence of **barium.**

White precipitate ..... } presence of **barium.**
$BaFl + SiFl^2$.

The filtrate, **F. 24**, is mixed with solution of sulphate of soda, left for an hour, and then boiled.

No precipitate..... absence of **strontium.**

White precipitate ..... } presence of **strontium.**
SnO, SO^3.

Note.—This precipitate should be tested before the blowpipe, to confirm the indication of strontia by the production of the characteristic red-coloured flame.

The filtrate, **F. 25**, is mixed with ammonia and oxalate of ammonia solution.

No precipitate..... absence of **calcium.**

White precipitate ..... } presence of **calcium.**
$CaO, \dot{O}$.

The filtrate, **F. 23,** or the solution that has been tested with carbonate of ammonia, is mixed with phosphate of ammonia solution, and kept warm for some hours.

No precipitate..... absence of **magnesium.**

White precipitate ..... } presence of **magnesium.**
$2MgO, NH^4O, PO^5$. }

Note.—Before testing for magnesium, it is advisable to add a few drops of sulphate of ammonia solution, and if a precipitate is produced, to separate it by filtration, so as to be sure of the absence of barium and strontium.

The filtrate, **F. 26,** is evaporated to dryness, nitric acid added meanwhile, and the residue ignited, so as to separate ammoniacal salts, then dissolved in water, and the solution mixed with bichloride of platinum and alcohol.

No precipitate..... absence of **potassium.**

Yellow precipitate ..... } presence of **potassium.**
$PtCl^2KCl$. }

The filtrate, **F. 27,** is evaporated to dryness on a steam-bath, the residue ignited with some oxalic acid, then dissolved in water, and the solution mixed with antimoniate of potash solution.

No precipitate..... absence of **sodium.**

White precipitate ..... } presence of **sodium.**
NaO, SbO^5. }

Note.—When the substance under examination contains barium, strontium, calcium, magnesium, or phosphoric acid, it is advisable to prepare a portion of the filtrate, **F. 14,** to test for potassium and sodium, by mixing it with baryta solution and boiling, then separating baryta from the clear filtered liquid by means of carbonate of ammonia, evaporating the clear liquid to dryness, and dissolving the residue in water.

Examination for acids or electro-negative substances.

The acids of arsenic, antimony, and chromium would be detected in the examination for bases, &c.; the presence of carbonates, sulphides, or sulphites would also be indicated.

When the acids, &c. are combined with other substances than those belonging to the groups 4 and 5, these must be separated from the solution, either by boiling it with carbonate of soda, or by means of sulphuretted hydrogen, or

sulphide of ammonium, according to the nature of the substances to be separated.

In the case of substances soluble in acids but not in water, the solution must be effected by means of nitric acid, unless the preliminary examination shows the presence of organic acids, chlorides, bromides, iodides, or cyanides, in which case the substance should be boiled with carbonate of soda solution, the clear liquid mixed with nitric acid, and then neutralized with ammonia.

An opinion as to the acids, &c. present may generally be formed from the relation of the substance to solvents and the nature of the bases, &c. which it contains.

In all instances the determination of the presence or absence of organic acids in the preliminary examination, must be carefully attended to.

The neutral solution, containing ammoniacal salts, is mixed with nitrate of baryta solution.

No precipitate..... { absence of all the members of the first group.

White precipitate ..... { may contain one or more members of the first group.

BaO,SO^3; $2BaO,HO,PO^5$; BaO,SiO^3; BaO,SO^2; BaO,CO^2.

Note.—When the solution contains a sufficient amount of ammoniacal salts, the precipitate, **P. 41**, will not contain borate, oxalate, tartrate, or citrate of baryta, as might be the case in the absence of ammoniacal salts.

Fig. 67.

The precipitate, **P. 41**, is collected upon a filter, washed, and the filtrate, **F. 28**, set aside for subsequent examination.

The precipitate, **P. 41**, is transferred to a small flask, fig. 67, and hydrochloric acid poured upon it through the funnel, while the delivering-tube dips into baryta water.

Complete solution ..... absence of **sulphates.**

Insoluble residue..... } presence of **sulphuric acid.**
BaO,SO^3. }

When hydrochloric acid is poured on the precipitate, **P. 41,**

No gas is evolved..... { absence of **sulphites** and **carbonates.**

Gas is evolved..... { presence of **sulphurous** or **carbonic acid.**

The gas evolved is passed into baryta water.

No precipitate..... absence of **carbonates.**

Precipitate..... } presence of **carbonic acid.**
BaO, CO^2.

The gas evolved is passed into a saturated solution of sulphuretted hydrogen.

No precipitate..... absence of **sulphites.**

White precipitate..... } presence of **sulphurous acid.**
S.

Note.—In the analysis of substances insoluble in water, the precipitate, **P. 41,** cannot contain carbonic or sulphurous acids, and these acids must be tested for with a separate portion of the substance as above directed.

The insoluble residue, **P. 42,** is separated by filtration, and the filtrate, **F. 29,** evaporated to dryness in a steam bath, the residue moistened with hydrochloric acid and covered with water.

Complete solution..... absence of **silicates.**

White residue..... } presence of **silica.**
SiO^3.

The insoluble residue, **P. 45,** is separated by filtration, and the filtrate, **F. 30,** mixed with ammonia and sulphate of magnesia.

No precipitate..... absence of **phosphates.**

White precipitate..... } presence of **phosphoric acid.**
$2MgO, NH^4O, PO^5$.

Note.—When the substance does not contain organic acids, and those above mentioned have been separated by nitrate of baryta, or proved to be absent, the only inorganic acid that remains to be tested for, besides the salt radicals, is boracic acid. For this purpose it is preferable to mix a separate portion of the substance with concentrated sulphuric acid and alcohol. The presence of boracic acid is indicated by the green colour of the flame.

The filtrate, **F. 28,** or the solution that has been tested

with nitrate of baryta, is mixed with nitrate of silver solution.

No precipitate..... { absence of all the members of the second group.

White precipitate ..... { may contain one or more members of the second group.

AgI; [AgS; Ag^2Cfy; AgCsy;] AgCl; [AgBr;] AgCy; [Ag^2Cfdy;] AgO,$\bar{O}$; AgO,$\bar{T}$; AgO,$\bar{C}$; AgO,$\bar{M}$; AgO,$\bar{B}z$; AgO,$\bar{S}u$; AgO,$\bar{A}$.

Note.—The substances indicated by brackets would not often be met with.

The precipitate, **P. 47**, is collected upon a filter, washed, and mixed with dilute nitric acid.

Complete solution ..... { absence of all members of 1st division of 2nd group.

Insoluble residue..... { may contain one or more members of the first division of the second group.

AgI; [AgS; Ag^2Cfy; AgCsy;] AgCl; [AgBr;] AgCy; [Ag^2Cfdy].

The residue, **P. 48**, is collected upon a filter, washed, and the filtrate, **F. 31**, set aside for subsequent examination.

The precipitate, **P. 48**, is digested with ammonia.

Complete solution ..... { absence of all members of 1st subdivision of 2nd group.

Insoluble residue..... { may contain one or more members of the first subdivision of the second group.

AgI; [AgS; Ag^2Cfy; AgCsy].

The residue, **P. 49**, is collected upon a filter, washed, and tested with sulphuric acid to confirm the indication of iodine.

The filtrate, **F. 32**, is neutralized with nitric acid.

No precipitate..... { absence of all members of 2nd subdivision of 2nd group.

Precipitate ..... { may contain one or more members of the 2nd subdivision of the second group.

AgCl; AgCy; [AgBr; Ag^2Cfdy].

The filtrate or solution, **F. 31**, is mixed with chloride of sodium solution sufficient to precipitate the silver, the clear liquid neutralized with soda, mixed with chloride of calcium solution, well shaken, and left for half an hour.

No precipitate..... absence of **borates, tartrates, oxalates.**

White precipitate ..... may contain **boracic, tartaric,** or **oxalic acid.**

The residue, **P. 50,** is collected upon a filter, washed, and the filtrate, **F. 33,** set aside for subsequent examination.

Note.—Boracic acid may be present in **F. 31.**

The residue, **P. 50,** is digested with cold solution of potash.

Complete solution ..... absence of **oxalates.**

Insoluble residue..... $CaO, \overline{O}$. presence of **oxalic acid.**

The residue, **P. 51,** is separated by filtration, and the filtrate or solution, **F. 34,** boiled.

No precipitate..... absence of **tartaric acid.**

White gelatinous precipitate ..... $2CaO, \overline{T}$. presence of **tartaric acid.**

The filtrate, **F. 33,** or the solution that has been tested with chloride of calcium, is boiled for ten minutes.

No precipitate..... absence of **citrates.**

White precipitate ..... $3CaO, \overline{C}$. presence of **citric acid.**

The precipitate, **P. 53,** is separated by filtration, and the filtrate, **F. 35,** or the solution that has been boiled with chloride of calcium, mixed with alcohol.

No precipitate..... absence of **malates.**

White precipitate ..... $CaO, \overline{M}$. presence of **malic acid.**

Note.—When the substance contains citric acid, the production of a precipitate by alcohol is not a positive indication of the presence of malic acid, because citrate of lime is to some extent soluble in water, and is precipitated by alcohol as well as malate of lime.

The filtrate, **F. 36,** or the solution that has been mixed with alcohol, is evaporated to separate alcohol, made perfectly neutral, and mixed with sesquichloride of iron.

No precipitate..... absence of **succinates** and **benzoates.**

Brown precipitate ..... presence of **succinic acid** or **benzoic acid.**

The precipitate, **P. 55**, is collected upon a filter, washed, heated with ammonia, the liquid filtered, the filtrate evaporated nearly to dryness, and a portion mixed with chloride of barium and alcohol.

No precipitate..... absence of **succinates.**

White precipitate ..... } presence of **succinic acid.**
BaO,$\overline{Su}$. }

The remainder of the filtrate, **F. 37**, is evaporated to a small bulk, and mixed with hydrochloric acid.

No precipitate..... absence of **benzoates.**

Crystalline precipitate .. } presence of **benzoic acid.**
$\overline{Bz}$. }

Nitric acid is best tested for as directed at page 237, in a portion of the solution, from which bases have been separated by carbonate of ammonia.

Acetic acid is tested for with sesquichloride of iron in a portion of the same solution.

Formic acid is tested for with nitrate of silver, and its presence is indicated by the separation of metallic silver after a few minutes, from the white precipitate first produced.

These acids may be obtained apart from others, by distilling a portion of the solution with dilute sulphuric acid, and may then be tested for in the distillate.

The following tables serve to point out, in a general way, the relation of compounds to various solvents. For this purpose they are divided into four classes:—

1. *Substances soluble in water; the solubility of sulphate of lime in* 430 *parts of water at* 60° *F., being taken as the extreme limit.*

2. *Substances insoluble in water, but dissolved by hydrochloric or nitric acids.*

3. *Substances dissolved by nitric acid, but not by hydrochloric acid.*

4. *Substances that are not dissolved by either hydrochloric or nitric acid.*

The number in the table opposite the symbol of a metal, or of a metallic oxide, and under that of a salt radical, or of an acid, indicates the class to which, as regards solubility, the compound of those two substances belongs.

ABLES showing the relation of various compounds to the principal solvents used in analysis.

	O	S	Fl	Cl	Br	I	Cy	As
	3	3	1	4	4 Sol. in HBr	3	4	?
	$\frac{Hg^2O\ 3}{HgO\ 2}$	4	$\frac{Hg^2Fl\ 3}{HgFl\ 1}$	$\frac{Hg^2Cl\ 2}{HgCl\ 1}$	$\frac{Hg^2Br\ 2}{HgBr\ 1}$	$\frac{Hg^2I\ 3}{HgI\ 2}$	1	?
	2	2	2	1	2	2	..	?
	2	3	2	1	1	3	..	..
	2	2	2	1	1	1	1	?
	2	2	1?	1	1	..	2	?
	2	?	..	1	$\frac{AuBr\ ?}{AuBr^3\ 1}$	2	$\frac{AuCy\ 4}{AuCy^3\ 1}$	?
	2	4	1	$\frac{PtCl\ 2}{PtCl^2\ 1}$	1	$\frac{PtI\ 4}{PtI^2\ ?}$	$\frac{PtCy\ 4}{..}$	?
	2 in part	2	1	1	1	?	..	2
	2	2	1	1	2	2	..	..
	2	2	1	1	2?	1	..	..
	2	2	1	1	1	1	4 Sol.in HCy	3
	2	2	1	1	1	1	4	3
	2	2	1	1	1	1	2	3
	2	3	1	1	1	1	3	?
	2	2	1	1	1	1	4	3
	2	4	2	1	1	?	2	?
	2	2	1	1	1	?	..	2
	2	2	2	1	1	1	2	?
	1	1	2	1	1	1	1	?
	1	1	2	1	1	1	?	?
	1	1	4	1	1	1	1	?
	2	1	4	1	1	1	1	?
	1	1	1	1	1	1	1	?
	1	1	1	1	1	1	1	?
	1	1	1	1	1	1	1	..

	AsO^3	AsO^5	SbO^5	CrO^3	SO^3	PO^5	SiO^3	SO^2	CO^2	$\overline{O}$	$\overline{T}$	BO^3	$\overline{C}$	$\overline{M}$	$\overline{Bz}$	$\overline{Su}$	$\overline{A}$	$\overline{F}$	NO^5	ClO^5
AgO	3	3	3	3	1	3	4	1	3	3	3	3	3	3	3	3	1	1	1	1
Hg^2O	3	3	3	4	3	3	?	[illegible]	3	3	3	..	3	3	3	2	1	1	1	1
HgO	3	3	4	2	1	2	?	..	2	3	3	..	3	1 in part	2	2	1	1	1	1
PbO	3	3	3	2	4	3	4	2	3	3	3	3	3	3	1	2	1	1	1	1
BiO^3	..	2	..	2	1 in part	2 in part	4	2	2	2	2	2	..	..	1	1 in part	1	1	1 partly	1
CdO	..	..	..	..	1	2	..	1	2	2	1	2	2	..	2	1	1	1	1	1
CuO	2	2	2	1	1	2	4	1	2	2	2	2	1	1	1	1	1	1	1	1
AuO^3	..	..	..	..	..	..	..	..	..	..	..	..	..	..	2	..	..	..	..	..
PtO^2	..	3	..	..	1	..	..	1	..	1	..	..	..	..	2	..	1	..	1	..
SnO	?	2	..	2	1	2	..	..	..	2	2	2	..	1	1	2	1	2	1	..
SnO^2	?	?	4	2	1	..	..	..	..	1	..	..	..	..	1	2	1	2	1	..
SbO^3	2	2	4	2	2	1 in part	..	2	..	2	1	..	..	..	1	..	1	1	..	..
NiO	2	2	2	2	1	2	2	1	2	2	2	2	1	..	1	1	1	1	1	1
CoO	2	2	1	2	1	2	2	2	2	2	1	2	1	..	1	2	1	1	1	1

Mn^2O^3	..	..	..	..	..	1	2 4	..	..	..	..	..	..	..	..	..	..	..	..	..
Ur^2O^3	..	2	2	2	1	2	..	2	2	..	..	2	..	..	2	2	..	..	1	..
FeO	2	2	1	..	1	2	2	2	2	2	1	2	1	1	1	1	1	1	1	..
Fe^2O^3	2	2	2	2	1	2	2	..	..	1	1	2	1	1	2	2	1	1	1	..
Cr^2O^3	..	2	..	2	1	2	..	1	..	1	1	2	1	..	2	2	1	1	1	..
Al^2O^3	2	2	..	2	1	2	2 4	2	..	2	1	2	1	2	1	1 in part	1	1	1	..
ZnO	..	3	2	2	1	2	2	1	2	2	2	2	2	1	1	2	1	1	1	1
BaO	2	2	2	2	4	2	2	2	2	2	2	2	2	1	1	2	1	1	1	1
SrO	2	2	..	2	4	2	2	2	2	2	2	2	1	1	1	2	1	1	1	1
CaO	2	2	2	2	1	2	2	2	2	2	2	2	2	1	1	1	1	1	1	1
MgO	2	2	..	1	1	2	2 4	1	2	2	1	2	1	1	1	2	1	1	1	1
NaO	1	1	1 sparing	1	1	1	1	1	1	1	1	1	1	1	1	1	1	1	1	1
KO	1	1	1	1	1	1	1	1	1	1	1 KO,2T sparing	1	1	1	1	1	1	1	1	1
NH^4O	1	1	1	1	1	1	1	1	1	1	1	1	1	1	1	1	1	1	1	1

CHAPTER XI.

DYE-STUFFS; TESTS OF THEIR PURITY, AND THE METHODS OF VALUATION.

THE methods of valuation applicable to dye-stuffs mostly furnish indications that are merely relative, and in the absence of a more intimate acquaintance with the chemical history of some dye-stuffs, those methods of valuation which are imitative of the dyeing operations as executed on the large scale are probably the most trustworthy.

I. EXAMINATION OF DYE-STUFFS.

Madder; the root of *Rubia tinctorum*.

The numerous chemical investigations of madder, although presenting great discrepancies in their special results, all tend to justify the inference, that—

1. Madder root contains a great variety of peculiar organic substances.
2. Some among these are coloured; two red substances having the chief technical importance.
3. When the ground root is exposed to conditions favourable to fermentation, certain changes take place in it, which have a marked influence upon its value as a dyeing material.
4. That madder differs considerably according to the country in which it is grown.

The substances usually mixed with madder for adulteration are,—

Saw-dust, almond-husks, bran, pine-bark, acacia-wood, log-wood, sandal-wood, and *spent madder*. Some of these substances may be detected by means of the microscope, or a powerful magnifying lens.

Sand, clay, brick-dust, ochre, &c. These substances are indicated by grittiness of the madder when chewed; by the rapid formation of a sediment when the madder is stirred with water, and by the large residue left after ignition.

The average amounts of ash in different kinds of madder, dried at 212° F., are:—

Alsace madder	7·20	per cent.
Avignon madder	8·75	„
Levant madder	9·80	„
Peeled and cleaned madder	5·00	„

The natural inequality in the value of madder as a dye, and the difficulty of detecting adulteration by direct methods, render its valuation highly desirable; but there are considerable difficulties in devising a method of valuation, applicable to this material, which would be at once simple and trustworthy; probably that which furnishes results that are the most valuable, is the—

Dye-test,

which coincides exactly with the operation of dyeing on a large scale. By the application of this test, the relative value of a sample of madder is estimated from the intensity of the colour communicated to a piece of mordanted cotton cloth, by a known quantity of the madder, as compared with the tints obtained, under the same conditions, in equal-sized pieces of cloth by the use of fractional or multiple quantities of madder the quality of which is known.

The test-cloths are prepared by dyeing them in madder baths, representing individually, from 1 to 10 grms. of the standard sample; and another piece of cloth is dyed in a bath prepared with 10 grms. of the madder to be examined

The decoctions of madder are made in a series of wide-necked glass vessels, of 2 or 3 pints capacity, labeled and arranged in a water-bath. Into each of these is introduced a piece of mordanted calico, 2 inches square, together with the weighed quantity of madder and 1 pint of water at 104° F. The temperature of the bath is then raised to 167° F., maintained at that point for an hour and a half, and then at the boiling-point for half an hour, after which the pieces of cloth are taken out, rinsed in cold water, and dried.—Each piece

is then cut in two, and the halves numbered in accordance with the quantity of madder used for dyeing them.

The pieces of one series are immersed for half an hour in a bath consisting of 40 grains of soap dissolved in 2 pints of water, then rinsed in cold water; immersed for half an hour in a bath containing 8 grains of protochloride of tin, again rinsed in cold water, and dried. The relative value of the madder examined is then ascertained by a comparison of the cloth dyed by it, with the pieces dyed by different quantities of the standard sample.

The experiment may be made in a similar manner with skeins of yarn mordanted and prepared with oil for dyeing Turkey-red.

Direct estimation of the colour-substances.

Another method of estimating the value of madder, is to ferment a known quantity with water and yeast at a temperature of about 75° or 80° F. for twenty-four hours. A small quantity of acetic acid should be added so as to facilitate the conversion of the alcohol produced into acetic acid, and thus to prevent the solution of colour-substance by alcohol. When the fermentation has ceased, the liquid is poured off, and the residual madder boiled with acetic acid (1·012 sp. gr.) twice, the liquid filtered, evaporated to dryness, the residue dried at 212° F., and weighed; or,—

The madder may be charred with an equal weight of sulphuric acid, the mixture diluted with water, washed until the filtrate has no longer any taste, fatty substances extracted by means of a mixture of ether and alcohol from the residue, which is then boiled with alcohol (0·843 sp. gr.) twice, the liquid distilled, and the residue dried at 212° F., and weighed.

This method of valuation is applicable to garancine also.

Colorimetric test.

This consists in a comparison of the intensity of the colour of a decoction of the madder to be examined, with that of a decoction of a standard sample.

The instrument used for this purpose is called a colorimeter. It consists of two or more glass tubes half an inch

wide, rather more than a foot long, closed at the lower end, and fixed side by side upon a stand. At about five inches from the bottom there is a scratch upon each of the tubes, and in the upper part, an equal capacity is indicated by another scratch, the space between the two marks being divided into 100 equal parts, the lower mark serving as the zero-point.

To estimate the relative value of samples of madder, the tubes are filled up to the zero-point with decoctions, representing equal quantities of the several samples, and the darker-coloured decoctions diluted until they present the same intensity of colour as the others. The quantity of water required for this purpose is read off on the graduated part of the tubes, and the colour-value of the several samples will be directly proportionate to the amount of dilution requisite to produce equality of colour.

The madder to be examined and that which is to serve as a standard of comparison are dried at 212° F., and 25 grms. of each digested with ten times the weight of water at 68° F., the liquid poured off, the residues again digested with water, washed, dried, and weighed. The loss of weight represents sugar and mucilage.

A weighed portion of each of these residues is introduced into a flask and boiled for fifteen minutes with a solution of 30 grms. of alum in 200 grms. of water, the liquid poured off and the operation repeated twice with a fresh quantity of alum solution. The solutions thus obtained will contain the colour-substance of the several samples of madder, and the intensity of colour is estimated for each by means of the colorimeter.

Cochineal; the dried carcasses of *Coccus cacti*.

Several varieties of cochineal, differing much in value, are met with in commerce.

The substances with which it is sometimes adulterated, are—

White lead or *talc*. These substances are attached to the surface of the cochineal as an incrustation. They are indicated when a quantity of the suspected cochineal is rubbed between the hands, by the separation of a white powder which may be further tested with reagents.

Fragments of lead; indicated when the cochineal is powdered, by the metallic appearance of the flattened fragments.

Factitious cochineal; indicated when the substance is steeped in water, by the fragments falling into paste. This factitious cochineal is sometimes made of spent cochineal faced with talc.

The methods of valuation applicable to cochineal are of three kinds, viz. the direct estimation of the amount of colour-substance by precipitation, the indirect estimation by means of a bleaching substance, and the dye-test.

Direct estimation of the colour-substance.

This consists in precipitating the colour-substance, in combination with alumina, by means of a liquid containing a known amount of suspended alumina.

The test-liquid is prepared by dissolving alum in 32 parts water, and adding to the solution, ammonia just sufficient to precipitate the alumina. This liquid is preserved for use in a stoppered bottle, and must be well shaken each time it is used.

The instrument used for the experiment is a glass cylinder, 24 inches high and nearly 1 inch wide, which is graduated so as to furnish indications that may be compared with a sample of cochineal of known quality. For this purpose the colour-substance is extracted from a portion of the standard sample by digesting the powder with water, in a water bath, evaporating the solution to dryness, dissolving the residue in warm alcohol, and evaporating the clear liquid to dryness; 7 grains of this residue is placed in the glass cylinder, water added to the height of 3 inches, and a scratch made on the cylinder at this level which serves as the zero-point. The test-liquid is then added until the whole of the colour-substance is precipitated, and the liquid appears colourless. The level at which the liquid stands in the cylinder is marked, and the space between this mark and the zero-point divided into 70 equal parts.

In order to estimate the relative value of a sample of cochineal, 10 grains of the powder are completely extracted with 10 parts of hot water, the solution decanted from the sediment into the cylinder, and this operation repeated as

often as may be necessary. The volume of the solution is then made up with water to the zero-point, and the test-liquid added until the whole of the colour-substance is precipitated and the liquid appears clear. The number of degrees of test-liquid used will then indicate the value of the cochineal as compared with the standard sample.

Indigo; impure colour-substance of *Indigofera tinctoria*.

The valuation of indigo is best effected volumetrically by means of a bleaching substance. Chlorate of potash mixed with hydrochloric acid is most suitable for the purpose.

The test-solution is prepared by dissolving 2·50 grm. dry powdered chlorate of potash in water, and making up the volume to a litre.

In order to estimate the relative value of a sample of indigo, 1 grm. of the powder is covered with 8 or 10 parts of concentrated sulphuric acid, in a porcelain basin of 2 pints capacity, left for six or eight hours, and frequently stirred meanwhile. About 50 grm. of hydrochloric acid and 1½ pint of water are then added, the mixture heated to the boiling-point, and water added from time to time to compensate for evaporation. The test-solution is then added, from a graduated tube, until the blue colour is completely discharged. Between each addition of test-solution, the liquid should be stirred and brought to the boiling-point. The colour passes at first from blue to green, then acquires a brownish tinge, and lastly reddish brown, at which point the further addition of test-solution must be very cautiously made.

Ultramarine.

The best method of estimating the colour-value of ultramarine, is to mix a known quantity with six times the weight of powdered chalk, sulphate of baryta, white lead, or zinc white, and compare the colour of the mixture with that of a similar mixture containing ultramarine of known quality; or the relative value of several samples may be ascertained from the proportions of ultramarine requisite to give the same intensity of colour to equal weights of a white powder.

II. Examination of dyed or printed fabrics.

The diversity in the behaviour of colour-substances which are met with in dyed or printed fabrics, is so great that a

tabular arrangement of all the reactions which should be taken into account for ascertaining the dye-stuff with which any particular colour has been produced, would be almost impossible.

In deciding such a question it is necessary, in the first instance, to determine the nature of the material dyed, since, for various chemical or economical reasons, many colours are specially applied to one kind of stuff, and never, or very seldom, to any other.

Incineration of the cloth upon platinum foil furnishes a means of ascertaining what mordant has been employed, and the behaviour of the cloth while burning, indicates whether it consists of wool, silk, cotton, or flax fibre. The latter two give a much lighter ash than the others.

In the following table, the column stating the behaviour when burnt, is the only one which requires some explanation. The appearance of the ash is a sufficient indication whether a mordant has been used or not. In the former case, the ash is much more bulky, heavier, and frequently forms a kind of skeleton, exactly resembling the fibre, while the ash of stuffs which have not been mordanted, is very small in quantity, light, loose, and frequently melts to an almost imperceptible spot upon the platinum, sufficient in most instances to give a blue colour to a slip of moistened litmus. The mineral substances with which the various fibres are impregnated, for the purpose of dyeing, are—

Alumina; peroxide of iron; oxide of tin (most frequent); oxides of lead, copper, chromium; arsenous acid; sulphuret of arsenic.

Sometimes two of these substances are used together.

The residue left after incineration, may be examined more minutely in the manner described in Chapter X.

It is important to observe whether the colour has been communicated to the fabric by dyeing or printing, and in the latter case what other colours it may be associated with, because the presence of one colour frequently excludes the possibility of some other colour being used in the same piece, and thus limits the examination. For the incineration of printed fabrics, the different coloured portions should be cut out and burnt separately.

Name of the colour.	Incineration upon platinum foil.	Moistened with lemon juice or dilute hydrochloric acid.	Steeped in caustic soda.	Steeped in chloride of lime.	Special remarks.
1. Indigo blue. Vat blue; English blue; Saxon blue. *Indigofera tinctoria.*	Ash small in quantity, white and porous. Sometimes contains a little binoxide of tin.	Colour unaltered.	Colour unaltered. Saxon blue yields much colour.	Colour discharged.	Indigo blue is applied to woollen, cotton, silk, and linen stuffs. The vat blue is much superior to the Saxon blue which is frequently used in dyeing wool and silk, and does not resist the action of caustic alkalies.
2. Prussian blue. (Chemical blue.)	Leaves a reddish ash which gives the reaction of peroxide of iron.	Unaltered.	Colour discharged. Becomes yellow finally.	Unaltered or perhaps rendered darker and brighter.	Applied to silk, wool, and cotton stuffs. It resists the action of acids and of light colours, but not of soap.
3. Logwood blue. *Hæmatoxylon campeachianum.*	The border of the flame is green. The ash is whitish or grey, and contains alumina and oxide of copper.	Produces red or orange and reddish-blue spots; the hydrochloric acid becomes reddish-blue.	Becomes deeper coloured and clearer for a while.		
4. Ultramarine.	Residue blue; unaltered.	Colours discharged, with evolution of sulphuretted hydrogen.	Unaltered.	No great change.	Employed only in printing. Easily recognizable by the peculiar colour.

RED COLOURS.

Name of the colour.	Incineration upon platinum foil.	Boiled with water containing $\frac{1}{200}$ of soap.	Moistened with strong solution of potash.	Moistened with tin salt, strong hydrochloric acid, and water in equal parts.	Steeped in caustic ammonia.	Moistened with lemon-juice.	Special remarks.
Cochineal red. Lac-red presents very similar characters. *Coccus cacti.*	Ash contains either oxide of tin or alumina, or both.	The intensity of the colour is reduced, and the liquid acquires a dark red colour.	Produces brown spots, which are removed by vinegar.	The colour is partially discharged.	Renders the colour paler, and dissolves a portion.	Produces yellow spots, that are not entirely removed by ammonia.	Used for wool, silk, and cotton.
Brazil-wood red. *Cæsalpina echinata.*	Ash contains a small amount of alumina and oxide of tin.	The intensity of the colour is reduced, and the liquid acquires a dark red colour.	Produces purple spots, which are removed by vinegar.	Produces purple spots.	Produces a bluish tinge.	Produces yellow-red spots, that are removed by ammonia.	Used for wool, cotton, and silk.
Madder red. *Rubia tinctorum.*	Ash contains alumina.	Colour becomes brighter; after a while paler.	The soaped cloth becomes darker coloured.	No change.	No change.	No change.	Used for wool, cotton, and linen.
Turkey-red may easily be recognized by the peculiarity of the colour, and by its being less altered by reagents than any other red colour.—Turkey-red produced with Brazil-wood becomes brown with hypochlorite of lime, and is afterwards discharged.							
Safflower red. *Carthamus tinctorius.*	Ash does not indicate any mordant.	Colour extracted.					Used for silk; less frequently for cotton.

Name of the colour.	Incineration upon platinum foil.	Moistened with hypochlorite of lime.	Moistened with protochloride of tin and hydrochloric acid.	Moistened with lemon-juice.	Special remarks.
Catechu brown. *Acacia catechu* or *Nauclea gambir.*	Ash green; contains oxide of chromium, and generally oxide of copper; sometimes oxides of manganese or iron.	Colour slowly discharged.	Becomes dull.	Little alteration, sometimes becomes paler.	Resists the action of soap.
Manganese brown.	Ash contains manganese.	No change.	Colour discharged.	Colour discharged.	
Madder brown.	Ash contains oxide of iron and alumina.	Produces pink spots.	Becomes yellow-red, almost orange.	Produces pale brown spots, that are removed by ammonia.	
Dye-wood brown. Various dye-stuffs with iron mordant.	Ash contains oxide of iron; sometimes oxide of copper and oxide of tin.	Produces rust-coloured spots.	Colour discharged.		

YELLOW COLOURS.

Name of the colour.	Incineration upon platinum foil.	Boiled with water containing $\frac{1}{200}$ of soap.	Moistened with strong solution of potash.	Moistened with tin salt, strong hydrochloric acid, and water in equal parts.	Steeped in caustic ammonia.	Special remarks.
Quercitron yellow. *Quercus tinctoria.*	Ash contains alumina.	Liquid becomes coloured without much injuring the colour.	Produces brownish spots, which become green with vinegar.	Renders the colour sulphur-yellow.	While moist it appears brownish; after drying unaltered.	A tolerably fast colour for wool, little used for silk, but much for cotton and linen. Dyes only the mordanted parts.
Sap yellow. *Rhamnus infectoria.*	Ash contains alumina; sometimes tin.	Liquid becomes coloured without perceptibly extracting colour from the stuff.	Produces brownish-yellow spots.	Orange-yellow spots.	Alters the tone but little, dissolves some.	Used only in printing.
Fustic yellow. *Morus tinctoria.*	Ash contains alumina.	Nearly decolorized.	Becomes somewhat darker; acetic acid restores the colour.	Produces sulphur-yellow.	Produces dark orange; the liquid also becomes sulphur yellow.	Used for wool more than for cotton. Becomes red with nitric acid.
Weld or woad yellow. *Reseda luteola.*	Ash contains alumina; sometimes traces of copper and tin.					Used for wool, silk, and cotton.

Chromates of lead.	soda upon charcoal gives lead globules.			borders.		
Orpiment yellow. *Sulphide of arsenic.*	Volatilizes with fumes and evolves odours of sulphurous acid and arsenic.					Used for wool, silk, and cotton; not so much as formerly.
Nankeen yellow. *Hydrated oxide of iron.*	Ash contains iron.	Becomes rather brighter.	Becomes rather darker.	Produces white spots.	Unaltered.	Hydrochloric acid and ferrocyanide of potassium produce blue spots.
Turmeric yellow. *Curcuma longa.*		Becomes brownish; colour restored by weak acids.	Becomes brownish; colour restored by weak acids.		Becomes brownish.	Used together with other dyes such as weld, cochineal, &c., generally for silk and wool.
Annotto yellow. *Bixa orellana.*	Becomes greenish-blue with concentrated sulphuric acid.					Used more for silk than cotton.
Picric acid.	Colour discharged by alkalies.					

GREEN COLOURS.

Name of the colour.	Incineration upon platinum foil.	Moistened with hypochlorite of lime solution.	Moistened with caustic alkali.	Moistened with lemon juice.	Moistened with tin salt and hydrochloric acid.	Special remarks.
Arsenite of copper.	Ash contains copper.	No change.	Becomes black when immersed and heated.	No change.		Used chiefly in printing, and but seldom.
Chrome green.	Ash green; contains oxide of chromium.	No change.	No change.	No change.		Used only in printing.
Chrome green.	Ash red or yellow; contains lead.	No change.	Becomes yellow.	No change.	Becomes blue.	
Indigo blue and **Vegetable yellow.** a. *Vat blue.* b. *Indigo.*	Ash contains alumina.	Colour discharged and not restored by ammonia.	Becomes olive-coloured for a while. Colour extracted by caustic soda.	Becomes paler.		
Prussian blue and **Vegetable yellow.**	Ash reddish; contains oxide of iron.	Becomes blue.	Becomes yellow.	Becomes rather paler.		Seldom used in dyeing.

Vegetable yellow.	of copper.			entirely restored by ammonia.		
Chrome yellow and **Indigo blue.**	Ash yellow; contains lead.	Becomes yellow.	Becomes blue.	No change.	Becomes blue, and after a while the colour is discharged.	Seldom used in dyeing.

BLACK AND GREY COLOURS.

Name of the dye.	Incineration upon platinum foil.	Moistened with hydrochlorite of lime.	Moistened with tin salt and hydrochloric acid.	Moistened with lemon-juice.	Boiled with water containing $\frac{1}{200}$ of soap.	Special remarks.
Logwood black with *iron mordant.*	Ash contains oxide of iron, sometimes oxide of alumina and oxide of copper.	Discharged very slowly.	Acquires a violet tinge.	Produces slight spots, that are removed by ammonia.	Little alteration.	Moistened with hydrochloric acid, the dye rubs off with a reddish colour upon white paper.
Nut gall black with *iron mordant.*	Ash contains iron.	Produces white spots.	Produces white spots.	Produces white spots, that become reddish-yellow with ammonia.	Moderately permanent.	
Chrome black.	Ash greenish; contains oxide of chromium.	Becomes chestnut-brown.	Produces dirty violet spots.		Little alteration.	
Madder black or **Garancine black.**	Ash reddish; contains iron, and frequently alumina.	Becomes reddish-brown, yellow, and is finally discharged.	Becomes rusty-brown; the moist spots do not colour paper.	No change.	No change.	Used only for printing.
Black upon *vat blue.*		Discharged.	Produces blue spots, that become yellow with	Produces blue spots.	No change.	Used for wool.

Name of the colour.	Incineration upon platinum foil.	Moistened with hypochlorite of lime solution.	Moistened with lemon juice.	Boiled with water containing $\frac{1}{200}$ of soap.	Moistened with potash solution.	Special remarks.
Madder violet. *Rubia tinctorum.*	Ash contains iron.	Becomes nankeen-yellow.	Produces yellow spots, removed by ammonia.	Colour slightly reduced; becomes bluer.	Produces violet spots, removed by ammonia.	
Alkanet violet. *Anchusa tinctoria.*	Ash contains alumina.	Little change.	No change.	Acquires a lilac tinge.	Produces blue spots, removed by vinegar.	Fades under the influence of light.
Logwood violet. *Hæmatoxylon campeachianum.*	Ash contains alumina.	Becomes nankeen-yellow.	Produces red spots, removed by ammonia.	Becomes rather more intense.	Produces brown spots, removed by vinegar.	Resists the influence of light tolerably well.

CHAPTER XII.

OILS, FATS, ETC.; SOAP, VOLATILE OILS, AND MATERIALS USED FOR LIGHTING; THE TESTS OF THEIR PURITY, AND THE METHODS OF VALUATION.

THE substances to which this chapter relates are all products of plants or animals, and consist either of compounds of fixed fat acids with organic or inorganic bases, or of mixtures of different compounds of carbon and hydrogen.

I. EXAMINATION OF OILS AND FATS.

It may be taken as a general rule in reference to the examination of oils, that although there may be physical characters or chemical reactions, by means of which certain oils may be distinguished from others with tolerable certainty, still when it is necessary to test for adulteration, all general characters are totally fallacious.

The density of some oils has been considered characteristic, and the fact that the density of oils differs only within very narrow limits, was sought to be met by the use of very delicate hydrometers, such as those of Fischer, Gobley, and others; but the use of delicate instruments is attended with serious difficulties, and hence a proposition has been made by Laurot to conduct the experiment at 212° F. However, none of these methods have been found adequate, because the variation in the density of a particular oil, according to its age, mode of preparation, &c., is frequently quite as great as the difference between it and the density of some other oil which is used for adulteration. This want of uniformity is evident from the following table:—

	Scharling.	Lefèbre.	Schübler.
Rape oil (winter)...	0·9228	..... 0·9154	..... 0·9128
Linseed oil	0·9383	..... 0·9350	..... 0·9347
Poppy oil (old).....	0·9630	..... 0·9253	..... 0·9243
Fish oil.....	0·9175 to 0·9317	..... 0·9240	..... 0·9231
Olive oil	... ...	..... 0·9180	..... 0·9176

Moreover oils do not expand equally when heated, so that an oil which at 60° is heavier than another, may at 212° F. be much lighter. Hence it appears that the determination of density as a means of detecting the adulteration of fat oils is applicable only in a very few instances, and is generally inadequate for the purpose.

As a preliminary test it is frequently advantageous to heat a few drops of the oil in a porcelain capsule, by which means the peculiar odour is rendered far more distinct, and the presence of admixtures may sometimes be detected in this way.

Besides the adulteration with other oils of inferior value, oils are sometimes mixed with the following substances:—

a. *Colophony or other kind of resin*; indicated by a white clotty precipitate on the addition of an alcoholic solution of acetate of lead, to the clear liquid obtained by shaking and boiling the oil with alcohol 0·88 sp. gr.

b. *Oleic acid* (obtained in the preparation of stearic candles); indicated, even in oils which have become rancid, by the acid reaction upon moistened blue litmus. The litmus paper should be dried by pressure between bibulous paper after being dipped into the oil. The acid reaction in such a case may however be owing to a trace of sulphuric acid introduced in the rectification of the oil.

c. *Sulphuric acid, lead, alum* are met with in many oils; the tests for their detection are described at p. 320.

Olive oil; the produce of varieties of *Olea europæa*.

The most probable adulteration is,—

Poppy oil; indicated when the oil is shaken, by the production of a froth.

Rousseau having observed that pure olive oil is a very feeble conductor of electricity, and that when mixed with poppy oil it conducts much more readily, proposed for this purpose the use of an instrument called a diagometer, constructed so as to show the electrical conductibility of the oil to be examined; but the diagometer does not furnish trustworthy indications, for Soubeiran and Blondeau have found that many kinds of olive oil conduct as well as olive oil containing 5 per cent. of poppy oil. These oils, although unadulterated, are mostly inferior in quality, being obtained from fermented olives.

Poutet employs a solution of six parts mercury in 7·5 nitric acid (1·356 sp. gr.) without the aid of heat. The oil in question is mixed with one-twelfth of this solution, shaken at intervals of ten minutes for two hours, and then allowed to rest for a day. After this treatment olive oil appears perfectly solid when lightly tapped upon its surface with a glass rod. When it contains 5 per cent. of poppy oil, the consistence of the mass is not, at the utmost, greater than that of tallow; with 10 per cent. about the same as lard.

Soubeiran and Blondeau have tried this test among others, and they admit the perfect certainty of its indications for olive oil containing 10 per cent. of poppy oil, as well as the possibility of detecting 5 per cent., although with less certainty. However, the method is inapplicable to quantitative valuation.

Boudet, and subsequently Fauré, have modified Poutet's test; the former, ascribing the action of the solution to the presence of hyponitrous acid, proposed treating the oil with 4 per cent. of a mixture consisting of 3 parts nitric acid (1·319 sp. gr.) and 1 part hyponitrous acid (1·255 sp. gr.) —prepared by distilling 1 part of starch with 8 parts of nitric acid at a very gentle heat—shaking and allowing it to rest at 50° F. until the oil becomes so solid that the vessel may be turned upside down without its running out, and to estimate the purity of the oil from the time which elapses between the mixing and the degree of the solidification.

Soubeiran and Blondeau found that pure olive oil, treated in the same manner, always solidifies sooner than the same oil mixed with poppy oil. By experiments with twenty-five samples of pure olive oil, differing in age and source, they found that solidification took place in from 45 to 49 minutes; with 10 per cent. of poppy oil from 48 to 97 minutes. Consequently this method of testing is as defective as the former, for the differences that exist among the unadulterated olive oils are too great to admit of any accurate results being obtained by means of it. Soubeiran and Blondeau likewise found that ammonia, which when shaken with some oils forms a doughy and with others a clotty mass, fails to give any trustworthy indications when applied to mixtures of the oils.

The inferior viscidity of pure olive oil as compared with oil adulterated with poppy oil, may afford some approximative indication of its purity, and the test is very simple. When a small quantity of pure olive oil is shaken in a test-tube, the air-bubbles are found to disappear very quickly. But this is not the case with oil containing poppy oil. With 10 per cent. of the latter oil, the bubbles disappear very slowly, gradually flattening at the surface; with 5 per cent. of the latter oil, the difference is still perceptible, but is not so decisive. This test does not admit of any great accuracy, but it and that of Poutet appear to be preferable to the others.

The action of potash upon olive oil is recommended as a safe test of the goodness of olive oil for the purpose of dyeing Turkey-red. About 20 cubic centimeters of the oil are shaken with 10 times the volume of potash solution (1·03 sp. gr.) in a cylindrical glass vessel. The experiment is made comparatively, a sample of known good quality being taken as the standard. Several samples of oil may be tested at the same time, and they must all be equally shaken. Good olive oil treated in this manner gives a milky liquid with a strong persistent froth upon the surface; inferior kinds give a thinner, bluish or yellowish, translucent liquid and a light froth which soon disappears. After the mixtures have stood for twenty-four hours, they are examined. There should not then be any large oil-globules among the froth, and it should appear curdy; the liquid beneath should not be thin and bluish, all these characters being indicative of inferior oil. When the froth consists of small bubbles, and is tolerably persistent, and when the whole mass of liquid is white and mucilaginous, the oil is of good quality. Oils of intermediate value present appearances which exhibit more or less of both these extremes.

Maumené has quite recently made known a method which he states to be applicable for distinguishing all drying oils from those that do not dry, but his experimental data refer particularly to the adulteration of olive oil with poppy oil.

This method is based upon the different elevation of temperature which takes place when poppy oil is mixed with sulphuric acid, compared with that resulting from the

mixture of olive oil with the same acid, under precisely similar circumstances. The experiment is made as follows. 50 cubic centimeters of the oil to be examined is introduced into a beaker glass, and the temperature observed; 10 cubic centimeters of sulphuric acid at the same temperature are then added; the whole well mixed together, and the greatest elevation of the thermometer observed. With pure olive oil, the temperature rises 76° F. above the temperature of the liquids before mixing. With poppy oil, on the contrary, the increase of temperature amounts to 128° or 76° F.

Maumené considers that when a sample of olive oil, treated in this manner, gives an elevation of temperature amounting to more than 76° F., it is a sufficient proof of adulteration.

Rape oil.—According to Davidson, the adulteration of olive oil with rape oil, which is sometimes practised, may be detected by means of Poutet's test. When amounting to 10 per cent., the detection is easy, but the experiment should always be made comparatively.

Chevallier states that *honey* is sometimes mixed with olive oil; its presence is indicated, when the oil is shaken with water, by its solubility.

Stearine is indicated by the separation of sand-like granules at the bottom of the vessel when the oil is cooled to 50° F. Pure olive oil does not begin to solidify until cooled to 36°·5 F.; and then the granules float in the liquid oil.

Sulphuric acid is sometimes employed for the purpose of bleaching, or separating mucus from the oil, and is not always perfectly removed. Its presence is indicated by the acid reaction of water that has been shaken with the oil, and the precipitate which it gives with chloride of barium in the presence of free hydrochloric acid.

Oxide of lead.—Rancid oil is sometimes treated with litharge for the purpose of making it sweet and of decoloring it.—The presence of lead is indicated, when the oil is shaken with an equal volume of weak acetic acid and a few drops of nitric acid, by a white precipitate (PbO, SO^3) on the addition of sulphuric acid to the liquid, and by a black precipitate with sulphuretted hydrogen.

Alumina is employed for separating mucus from the oil. Its presence it indicated, when the oil is shaken with water

and a few drops of sulphuric acid, by a flocculent precipitate on the addition of ammonia to the concentrated liquid.

Almond oil; the produce of *Amygdalus communis*.

This oil is distinguished from olive oil, poppy oil, and nut oil by agitation with basic acetate of lead solution. Almond oil gives a white turbid liquid; all the other oils a yellowish liquid. It is sometimes adulterated with cheaper oils.

Rape oil; the produce of several species of *Brassica*.

The most probable adulteration is,—

Fish oil; indicated, when the oil is impregnated with chlorine, by a brown coloration.

According to Lefèbvre, fish oil, when mixed with rape oil, separates completely in a layer at the bottom when the mixture is allowed to stand for some days.

Heidenreich observed that a drop of sulphuric acid, mixed with a few drops of rape oil, produces effects different from those which result from the mixture of the same acid with fish oil under otherwise similar conditions. The experiment is made upon a glass plate laid upon white paper, with 10 or 15 drops of the oil and 1 drop of sulphuric acid. It is advisable always to make two experiments; in the first instance, to let the acid fall gently into the midst of the oil, and observe the gradual action, afterwards at once to stir the oil and acid together.

With pure rape oil a greenish-blue colour is developed round the drop of acid, and in the centre, a few yellowish-brown streaks appear. When the oil and acid are at once stirred together, the whole assumes a greenish-blue colour, which does not become brown until 5 or 6 times as much acid is added.

With fish oil there is at first a peculiar motion from the centre towards the circumference, and a red colour is developed, which gradually becomes brighter, and after 12 or 15 minutes, passes into violet. When the oil and acid are at once mixed together, the red and violet colours are rapidly developed without any tinge of green.

With linseed oil and oleic acid, a brown colour is developed round the drop of acid. When mixed together with the acid, the former becomes at first thickish, and more so upon the addition of more acid; this is not the case with

any other oil in the same degree. Oleic acid stirred with sulphuric acid becomes dark-brown.

By means of this test 10 per cent. or less fish oil can be detected in rape oil from the development of the red colour as well as the greenish blue, peculiar to rape oil. The amount of the adulteration may be approximately estimated by comparative experiments with known mixtures.

The above-mentioned indication afforded by the smell of the oil when heated, may be of some assistance in estimating the proportion of fish or linseed oil, and particularly oleic acid.

The behaviour of the oils with a solution of bicarbonate of potash does not furnish very characteristic results.

Palm oil; produce of the *Avoira dais*.

Not only is this oil sometimes mixed with other substances, but a perfectly fictitious substance is sometimes sold under the name of palm oil, and consisting of wax, tallow, lard, coloured with turmeric and scented with violet root. From such mixtures palm oil is distinguished by its solubility in acetic ether. The presence of turmeric is indicated by the brown colour developed with caustic soda solution.

Fish oil, train oil; the produce of various species of *Delphinus* and *Balena*.

Fish oil is often adulterated with an inferior kind of linseed oil of brown colour. Except when bleached linseed oil has been used, its presence is indicated, when the oil is agitated with an equal volume of alcohol (0·815 sp. gr.), by the greenish-yellow colour communicated to the alcohol.

Greasing oils for machinery are tested by means of various apparatus capable of indicating the degree of resistance which they oppose to the motion of the machinery. Arrangements for this purpose, devised by Thomas, Sinclair, Nasmyth, &c., are described in most works on machinery.

Butter. See Chapter XIV.

In order to detect the presence of fat or oil in substances, the best method is to warm the substance with ether, and evaporate the liquid.

The estimation of the amount of fat in any substance may be effected in the same manner, the digestion with ether being, of course, repeated until the whole of the fat is extracted.

Wax; the produce of *Apis mellifica*.

The probable impurities and adulterations are,—

Tallow; indicated, when the amount is considerable, by the inferior consistence, and greasy feel of the wax; by the absence of the granular fracture, and by the taste when chewed. The distillate from wax, containing only 2 per cent. of tallow, gives, when shaken with water and filtered, a liquid from which acetate of lead throws down a precipitate, while this is not the case with pure wax.

Stearic acid; indicated, when the wax is boiled with lime-water, by a precipitate—stearate of lime—that remains suspended in the liquid, and by the neutralization of the lime.

Resin; indicated by the inferior brittleness of the wax, and by its adhesiveness. The resin is separated by alcohol.

Starch or *meal*, *earthy substances*, &c.; indicated, when the wax is dissolved in turpentine, by an insoluble residue, which is further examined to ascertain its nature.

In order to estimate the amount of stearic acid in adulterated wax, a known quantity is boiled for a few minutes with a solution of carbonate of soda in 50 parts of water, alcohol added until the liquid becomes clear, the insoluble portion separated by straining through linen, washed with water, melted, and weighed. The loss of weight represents stearic acid.

II. Examination and valuation of soap.

The probable admixtures, impurities, and adulterations are,—

Water; in very varying amount, which is indicated only by direct quantitative estimation.

Sand, pumice stone, clay, talc, sulphate of baryta, starch, &c.; indicated, when the soap is dissolved in alcohol, by a residue, which may be further examined to ascertain its nature.

In order to estimate the value of soap, several operations are requisite.

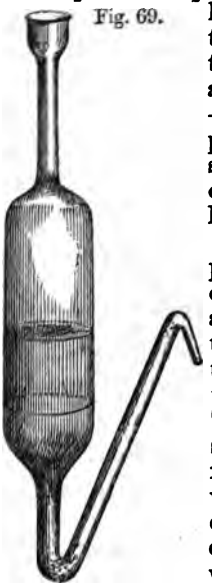
Estimation of water.—The soap, as thin shavings, is dried in a water-bath, and the diminution of weight observed; or a known quantity of the soap is boiled with a saturated solution of chloride of sodium, the mass dried, and weighed.

Fig. 68.

The separation of the last traces of water by drying is very difficult.

Estimation of the fat acid and alkali.—About 1 gramme of soap is weighed in a small beaker, fig. 68, and covered with ether and acetic acid. By this means the soap is decomposed; two layers of liquid are formed, the ether containing the fat acid or resin; the lower layer containing water, the alkali combined with acetic acid, free acetic acid, and the salts—chloride of sodium and alkaline sulphate—usually present in soap. Any insoluble admixtures remain at the bottom of the beaker, or suspended in the lower layer of liquid.

Fig. 69.



When the soap is completely decomposed, the whole mass of liquid is decanted into a large pipette, fig. 69, allowed to remain in the bulb until the two layers have separated completely, the beaker rinsed with ether and water, which are also poured into the pipette. Then by inclining the pipette, the water solution may be almost entirely separated from the ether solution, which is afterwards washed with successive quantities of distilled water, the separation being effected in the same manner. After washing the ether solution it is poured into a small weighed beaker, the adherent portion rinsed out of the pipette with a mixture of strong alcohol and ether, the whole evaporated to dryness upon a water-bath, and

the residue weighed several times until the weight ceases to diminish.

This residue of fat acid or resin may be further examined to ascertain its melting-point.

The water solution, together with the washings of the ether, containing the alkali, is evaporated to dryness in a small capsule, the residue ignited, and weighed. If desirable, this residue may be further examined, as directed in Chapter IX.

When the soap contains starch, gelatine, &c., the appearance of the residue left on evaporating the water solution to dryness, and its becoming charred when ignited, will afford sufficient indication of their presence. The amount of such substances may be ascertained approximatively by weighing the dry residue before ignition.

III. Examination of etherial oils.

The most frequent and probable adulterations are,—

Alcohol; indicated, when the oil is mixed with water in a graduated tube, by the diminution of the volume of the oil.

The presence of alcohol may also be ascertained by heating the oil with chloride of calcium, which becomes soft or liquid according to the amount of alcohol present, but remains unaltered when the oil is free from alcohol.

Acetate of potash may be substituted for chloride of calcium for the same purpose.

Fat oils; indicated by the viscidity of the liquid, by air-bubbles remaining at the surface after the oil has been shaken, and by the production of a persistent greasy spot upon paper, which is not removeable by alcohol.

Resin; indicated by the production of a greasy spot upon paper, that is removeable by alcohol.

Inferior volatile oils, turpentine; indicated, probably with most certainty when a drop of the oil is rubbed on the hands, by the smell. But the detection of admixtures of this kind is generally very difficult. Turpentine is in some cases indicated by the perfect solution of the etherial oil in question, by an equal volume of poppy oil.

Rose oil, attar of roses; the produce of *Rosa centifolia*, and *R. sempervirens*.

The substances with which rose oil is adulterated are, besides those already mentioned,—

Spermaceti; indicated by the higher temperature requisite for liquefaction.

Geranium oil; indicated, when the oil is mixed with concentrated sulphuric acid, by a peculiar unpleasant odour. The odour of pure rose oil is not affected by sulphuric acid.

Rose-wood oil; the produce of *Convolvulus scoparius*; indicated, when the oil is mixed with sulphuric acid, by a brown coloration; or when the oil is exposed in a watch-glass beside some iodine in another watch-glass, under a bell-jar, by the brown coloration or blackening of the oil.

A gelatinous substance impregnated with rose oil may be detected by warming the bottle.

Neroli oil; the produce of the flowers of *Citrus aurantium*.

Sometimes adulterated with—

Oil from the calyces and flower-buds of the orange-tree; which is said to be indicated, when sugar is immersed in the oil and dissolved in water, by the bitter taste of the solution.

Cinnamon oil and **cassia oil**; the produce of *Laurus cinnamomum* and *L. cassia*.

Sometimes adulterated with—

Clove oil; indicated, when the oil is dissolved in alcohol and mixed with a few drops of perchloride of iron solution, by a greenish coloration. Pure cinnamon oil gives a pure brown colour. The presence of clove oil is also indicated, when the oil is mixed with fuming nitric acid, by frothing and a reddish coloration; and when the oil is mixed with strong caustic alkali, by solidification. Pure cinnamon oil treated in a watch-glass gives off a mild fragrant vapour; the vapour of clove oil is acrid and excites coughing.

Bitter almond oil; the produce of *Amygdalus communis amara*. Boils at 348°·8 F.; density 1·043.

Sometimes adulterated with or substituted by—

Nitrobenzol; indicated by its insolubility in water, or when the oil is warmed with caustic potash, by a red coloration and production of a red crystalline distillate. Pure bitter almond oil treated with caustic potash, solidifies with production of benzoin and benzoate of potash. The density of nitrobenzol is 1·209; it boils at 415° F.

Alcohol; indicated, when the oil is mixed with twice its volume of nitric acid (1·50 sp. gr.), by evolution of nitrous acid vapour. Pure bitter almond oil treated in the same manner presents no reaction at first, but after two or three days is converted into yellowish crystalline benzoic acid.

Mineral naphtha.

Sometimes adulterated with—

Turpentine; indicated, when the oil is mixed with a few grains of iodide of potassium and water, by a yellow or orange coloration, and by the production of a crystalline substance when dry hydrochloric acid gas is passed into the oil at a low temperature.

Distilled waters sometimes contain copper and lead, which may be detected by evaporating to a small bulk and treating the residue with sulphuretted hydrogen.

IV. Valuation of oil, candles, gas, and other materials used for lighting.

The value of any material used for producing light can only be estimated relatively. The luminous effect of any substance depends upon—

1. The intensity of the light produced, and
2. The quantity of material consumed in its production;

so that it may be expressed numerically by the quotient of the luminous intensity, i, divided by the consumption of material, q, within a given time—

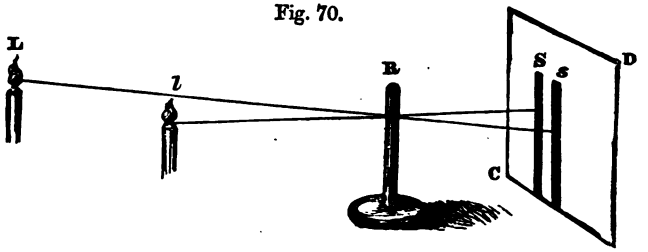
$$l = \frac{i}{q}.$$

The luminous intensity is estimated comparatively by means of the photometer, an instrument constructed upon the principle that the luminous effect is inversely proportionate to the square of the distance from the source of light. Thus when two sources of light produce respectively equal luminous effects at distances of 2 and 3 feet, the luminous intensity of the one would be 4, and that of the other 9.

The luminous intensity cannot be estimated directly, but only indirectly from the comparison of the shadows; the brighter light giving the darker shadow.

The photometer consists of a blackened iron rod, fig. 70, fixed upon a stand at some little distance from a wooden frame upon which a sheet of white transparent paper is

Fig. 70.



stretched. The lights to be examined are placed so that each throws a shadow of the iron rod upon the paper, and that the two shadows are near each other. The distances of the lights from the rod are then adjusted so that both the shadows appear equal, which may be best ascertained at the back of the screen from the simultaneous disappearance of the shadows when a lighted candle is brought near to it.

In such observations a definite and constant source of light, such as a wax candle, is employed as the standard of comparison. If, for instance, the squares of the distances at which the flame of a wax candle and that of a tallow candle, of the same size, produce equal shadows upon the paper screen, are respectively 14·6 and 13·2, the relative luminous intensities of the flames will be =100 : 90·41.

Then, if the consumption of wax is 9·56 and that of tallow 10·51 in the same time, the respective luminous effects will be,— *Wax.* *Tallow.*

$$\frac{14{\cdot}60}{9{\cdot}56} = 1{\cdot}527, \text{ and } \frac{13{\cdot}20}{10{\cdot}51} = 1{\cdot}256, \text{ or } = 100 : 82{\cdot}25.$$

Then to estimate the light-value, the price of the material must be taken into account, for if the prices of wax and of tallow were =7 : 3, the cost of producing the same luminous effect with wax and with tallow would be = 100 : 39·14, for—

$$7 \times 100 = 700 \quad \text{and} \quad 100 \times \frac{82{\cdot}25}{3} = 274.$$

In estimating the luminous effect of gas, the quantity consumed should be measured in cubic feet, and it must

be remembered that the luminous effect of gas varies according to—

1. The shape of the flame, depending upon the kind of burner used, and

2. The height of the flame, depending upon the pressure.

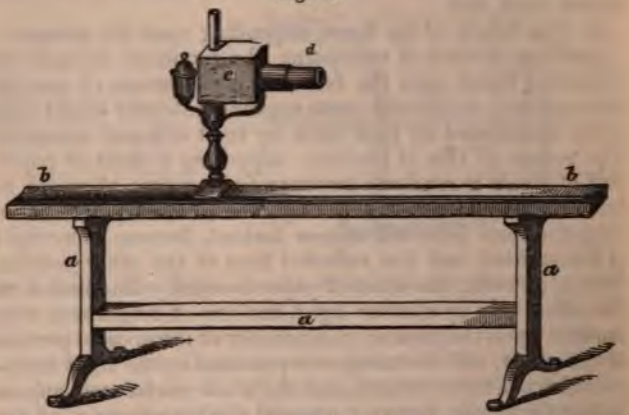
Another method of estimating the luminous intensity of a flame is based upon the fact that a diaphragm of unequal transparency at different parts appears uniformly bright only when illuminated on both sides by light of equal intensity. The reason of this is obvious: supposing a sheet of paper, half of which is rendered transparent by means of grease, to be illuminated on one side only, the greased portion seen from the same side, will appear darkest, because more light is transmitted, and less reflected than at the other portion. When, however, the sheet of paper is equally illuminated on the other side by another light, the loss of brightness, consequent upon the greater transparency of the greased portion of the paper, will be exactly compensated by the light transmitted from the other side, and the whole surface will appear uniformly bright, however unequal the transparency of the two halves may be. It also follows that when the light behind the paper is more intense than that in front of it, the greased portion will appear proportionately brighter; and when the light is less intense, it will appear darker than the portion that is not greased.

The photometer, fig. 71, used for estimating the value of lighting materials by this method, consists of a small tin box, *c*, blackened inside, and furnished with a sliding tube, *d*, and a lamp, L, or holder for a wax candle or gas-burner. The end of the sliding tube is covered with a diaphragm of white paper rendered uniformly translucent, by means of a solution of spermaceti in mineral naphtha, with the exception of a ring about six or eight lines diameter, at the centre. This box is attached to a metal bar, *b*, *b*, by means of a support, so arranged as to slide along the bar, which is graduated in feet and inches.

The light to be examined is placed in front of the diaphragm, and the box moved towards or from it until the difference between the brightness of the ring and that of the rest of the diaphragm disappears entirely; then the square of the distance at which this effect is produced gives

the relative luminous intensity of the flame as compared with that which serves as a standard of comparison.

Fig. 71.



The value of coal-gas may be estimated by a chemical method, based upon the action of chlorine upon heavy carburetted hydrogen (olefiant gas).

For this purpose the gas is mixed over water with an equal volume of chlorine in a glass cylinder, 12 inches long, $\frac{1}{3}$ inch diameter, the capacity of which is divided into 100 equal parts. The vessel containing the mixture is immediately covered with a tin sheath. After about five minutes the heavy carburetted hydrogen will have been condensed by combination with chlorine, and by exposing the remaining gas to light, the marsh gas is also condensed.

In reading off the amount of contraction of the mixture, allowance must be made for the absorption of chlorine by water, amounting in this case to about 1 degree of the cylinder, which is to be deducted from the volume representing the condensed gases.

The amount of contraction is then observed; and since there were equal volumes of both gases, and there is an equal condensation of both, the number of degrees by which it is represented expresses the per-centage of heavy carburetted hydrogen in the gas examined.

CHAPTER XIII.

ALCOHOL, SPIRITS, WINE, BEER, ETC.; THE TESTS OF THEIR PURITY, AND THE METHODS OF VALUATION.

THE characteristic ingredient of these and similar liquids is alcohol; the differences in colour, taste, smell, &c. which they present, arise from the presence of substances derived incidentally from the raw material from which they are produced, and varying according to the mode of production.

The individual value of these liquids does not in all instances depend merely upon the amount of alcohol they contain, but is to a great extent conventional.

Alcohol; spirit of wine, $C^6H^4O^2$, with varying amount of water.—Colourless liquid, with peculiar odour; density of the anhydrous substance 0·79381 at 60° F., boils at 173° F.; density of proof spirit 0·91984; boils at 81° F.

The probable impurities are,—

Fusel oil, indicated, when a glass is rinsed with the spirit and then emptied, by the smell of the portion adhering to the sides. Alcoholic liquids containing fusel oil give, when mixed with a few drops of nitrate of silver solution and ammonia, and exposed to direct sunlight, a reddish or black precipitate. This is not the case with pure alcohol, but, in the presence of other oily or organic substances, the same effect is produced. Or, the spirit may be mixed with $\frac{1}{60}$th of hydrate of potash, evaporated to dryness on a steam-bath, and the residue mixed with dilute sulphuric acid, when, if fusel oil is present, its odour will become perceptible.

Wood-spirit, or *naphtha*, indicated by the smell, by the lower boiling-point—anhydrous wood-spirit boils at 152° F. —and by the coloration of the liquid when digested with hydrate of potash.

Soap, alum, or *chloride of calcium*, would be detected by the usual tests, applied to the residue left on evaporation.

Estimation of alcohol.—The valuation of alcoholic liquids is, in many cases, effected by estimating the amount of alcohol they contain.

When the liquid does not contain any other substance than alcohol and water, the amount of alcohol is indicated by the density of the liquid, and the value is expressed in degrees having reference to a legalized standard or "proof spirit," consisting of—

	By weight.		*By volume.*
Alcohol....	49·24	.....	57·05
Water	50·76	.....	42·95
	100·00		100·00

In ordinary practice an hydrometer is used for determining the density of alcoholic liquids, and the per-centage of alcohol by weight or volume, or the commercial expression of its value is found by reference to a table (see Appendix).

When the liquid, in which the amount of alcohol is to be estimated, contains other substances than alcohol and water, as in the case of cordialized spirits, wine, or beer, it is necessary in the first instance to separate the alcohol by distilling a measured quantity of the liquid, and carefully condensing the vapour.

When the liquid contains only a small amount of alcohol, it is not necessary to distil over more than one-sixth part in order to obtain the whole of the alcohol, but when the amount of alcohol is considerable, the distillation must be continued until half or two-thirds of the liquid has passed over. The volume of the distillate is then accurately observed, its density determined, and, to ascertain the amount of alcohol in the liquid under examination, the amount of alcohol corresponding with the density of the distillate, is divided by 6, 2, &c., according as the distillate amounts to $\frac{1}{6}$, $\frac{1}{2}$, or other fraction of the liquid operated upon;—thus if the density of the distillate were 0·920 at 60° F., it would contain 57·05 per cent. by volume, and 49·24 per cent. by weight of alcohol, and if its volume amounted to one-sixth of the liquid operated upon, the amount of alcohol in the latter would also be one-

sixth of that in the distillate, or 9·508 per cent. by volume, and 8·206 per cent. by weight.

The apparatus best suited for this operation is represented by fig. 72. The condenser, B, is a square copper box, with a flat worm, A, of tinned copper, and two tubes, C C′, by means of which a current of cold water is passed through

Fig. 72.

the condenser. The front side of the condenser is supposed to be removed for the purpose of showing the interior arrangement.

One end of the worm terminates in a delivery pipe through which the condensed spirit runs into the receiver, F, and at

the other end there is a metal cap, D, with a flanged lining, G, fitting close upon a metal collar, C, cemented upon the neck of the distilling flask, which is connected with the worm by screwing the cap, D, upon the collar, C.

The examination of cordialized spirits, wine, beer, &c., will generally be limited to the determination of two points, viz.

1. The amount of alcohol; and
2. The presence or absence of injurious or fraudulent admixtures.

Cordialized spirits contain, as normal ingredients, besides alcohol and water, a variety of flavouring and colouring substances, and sometimes sugar.

The probable impurities, besides those already mentioned as likely to be present in raw spirit, are,—

Copper, *tin*, *lead*, *zinc*, originating from the vessels in which the spirits are distilled or stored; these substances would be indicated by the usual tests, in the residue left on evaporation.

Sulphuric acid would also be recognizable in the residue.

Ginger, *pepper*, *pimento*, &c. would be recognizable by the taste and smell of the extract left by evaporating the spirit to one-tenth its bulk.

Wine contains, as normal ingredients, besides alcohol and water, sugar, gum, and albuminoid substances, organic acids —chiefly tartaric acid—colouring and flavouring organic substances of various kinds, both fixed and volatile, the latter constituting the "bouquet."

Wine may contain, besides the ingredients proper to it, admixtures of—

Alcohol, *sugar*, *flavouring* or *colouring substances*, in greater or less amount, or it may be wholly factitious, but generally speaking there are not any means of ascertaining with certainty whether these substances have been added fraudulently or not.

The amount of alcohol in wine is estimated in the manner already described, by distilling a measured quantity and observing the density of the distillate, &c.

When the distillate contains acetic acid, it must be neutralized with carbonate of soda, and again distilled before the density is taken.

The amount of extract in wine is estimated in the same manner as the extract of beer, p. 336.

The amount of free acid in wine may be estimated volumetrically by means of a dilute alkaline solution.

The amount of free tartaric acid in the extract of wine is estimated in a weighed quantity of wine by adding an excess of a concentrated solution of neutral tartrate of potash, collecting the precipitate of bitartrate of potash, and weighing it.

Bitartrate of potash contains 70 per cent. of tartaric acid, and one-half of the quantity in the precipitate would be derived from the wine, the other half from the neutral tartrate of potash. The presence of other acids—citric, malic, and acetic acids—tends to augment the results thus obtained; but this is not of any account, as the tartaric acid preponderates over them so largely.

The amount of bitartrate of potash in wine is estimated by evaporating a portion until the residue becomes syrupy, then mixing it with alcohol (0·858 sp. gr.), filtering and washing with alcohol until the filtrate passes colourless. The insoluble portion is then burnt in a platinum basin, the carbonaceous residue extracted with hot water, the quantity of carbonate of potash estimated by means of a test acid, and the corresponding quantity of bitartrate of potash ascertained by calculation.

The amount of sugar in wine is estimated by fermenting the extract with beer yeast, distilling off the alcohol produced, estimating it as already directed, and calculating from the quantity obtained the corresponding quantity of sugar, upon the assumption that 100 parts absolute alcohol represent 59 parts of anhydrous grape-sugar. See also Chapter XIV.

Beer contains, as normal constituents, alcohol, sugar, dextrine, colouring and flavouring substances, and carbonic acid.

Besides these substances beer may contain,—

Acetic acid, indicated sufficiently by the smell and taste.

Soda or *potash*, added for the purpose of neutralizing the acid of beer that has become sour; tested for by evaporating the beer to dryness, burning the residue, and examining the ash in the usual manner; an addition of alkali is indicated when the amount of alkaline carbonate exceeds 5 grains in the pint.

The amount of alcohol in beer is estimated by distillation in the manner already described.

The amount of extract may be estimated approximatively, by diluting the residue left after separating the alcohol by distillation, until it measures exactly as much as the quantity of beer operated upon, then observing the density of the liquid, and referring to table XII. in the Appendix to ascertain the corresponding amount of extract.

Determination of the original gravity or density of beer-wort.

This is effected best by a modification of the method already described for estimating the amount of alcohol and of extract in beer. This method is now adopted by the Excise for the purpose of ascertaining the amount of drawback to be allowed upon beer exported.

It is based upon the fact that—

In beer-brewing, there is a uniform or nearly uniform relation, between the production of alcohol and that of extractive substance; and upon—

The direct estimation of the attenuation, or reduction of density of wort, consequent upon the conversion of sugar into alcohol.

The numerical values of this reduction of density corresponding to different amounts of alcohol have been determined experimentally, and are given in the following table A.

The amount of alcohol, or spirit indication of the beer, is expressed in degrees, which form the first column of the table, and the diminution of density corresponding to degrees and fractions of degrees of spirit indication, is expressed by the numbers opposite the degrees and under the fractions.

Then, by adding together, the value thus obtained for the reduction of density consequent upon the fermentation of the wort, and the extract gravity, or the observed density of the beer when deprived of alcohol and made up to the original volume, the density of the wort, or as it is technically termed, the original gravity of the beer, is found.

Thus if the spirit-indication is found by experiment to be 9·9, the loss of gravity, or attenuation, will amount to 43·7 degrees; and if the extract gravity of the beer is 1044·7, the original gravity would be 1088·4.

Degrees of spirit-indication with corresponding degrees of gravity lost for malt wort.

Degrees of spirit-indication.	·0	·1	·2	·3	·4	·5	·6	·7	·8	·9
0	··	·2	·6	·9	1·2	1·5	1·8	2·1	2·4	2·7
1	3·0	3·3	3·7	4·1	4·4	4·8	5·1	5·5	5·9	6·2
2	6·6	7·0	7·4	7·8	8·2	8·6	9·0	9·4	9·8	10·2
3	10·7	11·1	11·5	12·0	12·4	12·9	13·3	13·8	14·2	14·7
4	15·1	15·5	16·0	16·4	16·8	17·3	17·7	18·2	18·6	19·1
5	19·5	19·9	20·4	20·9	21·3	21·8	22·2	22·7	23·1	23·6
6	24·1	24·6	25·0	25·5	26·0	26·4	26·9	27·4	27·8	28·3
7	28·8	29·2	29·7	30·2	30·7	31·2	31·7	32·2	32·7	33·2
8	33·7	34·3	34·8	35·4	35·9	36·5	37·0	37·5	38·0	38·6
9	39·1	39·7	40·2	40·7	41·2	41·7	42·2	42·7	43·2	43·7
10	44·2	44·7	45·1	45·6	46·0	46.5	47·0	47·5	48·0	48·5
11	49·0	49·6	50·1	50·6	51·2	51·7	52.2	52·7	53·3	53·8
12	54·3	54·9	55·4	55·9	56·4	56·9	57·4	57·9	58·4	58·9
13	59·4	60·0	60·5	61·1	61·6	62·2	62·7	63·3	63·8	64·3
14	64·8	65·4	65·9	66·5	67·1	67·6	68·2	68·7	69·3	69·9
15	70·5									

TABLE B.—To be used in ascertaining original gravities by the evaporation method.

Degrees of spirit-indication with corresponding degrees of gravity lost for malt wort.

Degrees of spirit-indication.	·0	·1	·2	·3	·4	·5	·6	·7	·8	·9
0	—	·3	·7	1·0	1·4	1·7	2·1	2·4	2·8	3·1
1	3·5	3·8	4·2	4·6	5·0	5·4	5·8	6·2	6·6	7·0
2	7·4	7·8	8·2	8·7	9·1	9·5	9·9	10·3	10·7	11·1
3	11·5	11·9	12·4	12·8	13·2	13·6	14·0	14·4	14·8	15·3
4	15·8	16·2	16·6	17·0	17·4	17·9	18·4	18·8	19·3	19·8
5	20·3	20·7	21·2	21·6	22·1	22·5	23·0	23·4	23·9	24·3
6	24·8	25·2	25·6	26·1	26·6	27·0	27·5	28·0	28·5	29·0
7	29·5	30·0	30·4	30·9	31·3	31·8	32·3	32·8	33·3	33·8
8	34·3	34·9	35·5	36·0	36·6	37·1	37·7	38·3	38·8	39·4
9	40·0	40·5	41·0	41·5	42·0	42·5	43·0	43·5	44·0	44·4
10	44·9	45·4	46·0	46·5	47·1	47·6	48·2	48·7	49·3	49·8
11	50·3	50·9	51·4	51·9	52·5	53·0	53·5	54·0	54·5	55·0
12	55·6	56·2	56·7	57·3	57·8	58·3	58·9	59·4	59·9	60·5
13	61·0	61·6	62·1	62·7	63·2	63·8	64·3	64·9	65·4	66·0
14	66·5	67·0	67·6	68·1	68·7	69·2	69·8	70·4	70·9	71·4
15	72·9									

The spirit-indication may be determined in two ways; either:

1. As a difference between the density of water, and that of the spirit obtained by distilling the beer as above directed and making up the distillate with water, to the volume of the original beer; or:

2. As the difference between the density of the beer and that of the liquid that has been deprived of alcohol by boiling the beer and making up to the original volume with water.

The spirit-indication obtained by the former method is always somewhat higher than that obtained by the evaporation method, owing chiefly to the fact that the density of an alcoholic liquid containing sugar is proportionately greater than that of a similar liquid containing the same amount of alcohol without sugar, or, in other words, that alcohol reduces the gravity of sugar solution, or of malt wort less than it reduces that of water. Consequently, for ascertaining the original gravity of beer by means of the spirit-indication obtained by the evaporation method, it is necessary to have another table, in the construction of which this circumstance is taken into account. The numerical values of the reduction of density corresponding to the spirit-indication determined by the evaporation method, are given in table B.

The evaporation method is less operose, but also less accurate than the distillation method.

In determining the original gravity of beer, the amount of acetic acid present should not be overlooked, particularly in the examination of old or hard beer, for this acid is produced by the alteration of alcohol; 60 parts representing 46 parts by weight of alcohol, and therefore the experimental results must undergo a correction corresponding to the amount of acetic acid, which is estimated volumetrically by means of an alkaline test-solution.

The amount of alcohol equivalent to acetic acid found, and the corresponding degrees of spirit-indication are given in the following table C, and the amount thus found must be added to the spirit-indication obtained directly by the distillation or evaporation method before referring to the tables to find the degrees of gravity lost.

TABLE C, showing the per-centage of alcohol, and the spirit-indication corresponding to the acetic acid found.

Per cent. acetic acid.	·0	·1	·2	·3	·4	·5	·6	·7	·8	·9
0	—	·02	·04	·06	·07	·08	·09	·11	·12	·13
1	·14	·16	·18	·20	·21	·22	·23	·25	·26	·27
2	·27	·29	·31	·33	·34	·35	·36	·38	·39	·40
3	·39	·41	·43	·45	·46	·47	·48	·50	·51	·52
4	·52	·54	·56	·58	·59	·60	·61	·63	·64	·65
5	·65	·67	·69	·71	·72	·73	·74	·76	·77	·78
6	·77	·79	·81	·83	·84	·85	·86	·88	·89	·90
7	·90	·92	·94	·96	·97	·98	·99	1·01	1·02	1·03
8	1·03	1·05	1·07	1·09	1·10	1·11	1·12	1·14	1·15	1·16
9	1·15	1·17	1·19	1·21	1·22	1·23	1·24	1·26	1·27	1·28
10	1·29	1·31	1·33	1·35	1·36	1·37	1·38	1·40	1·41	1·42

Experiment.—For the purpose of determining the original gravity of a sample of beer, the most convenient quantity to be taken is 1500 grain measures at 60° F. This is shaken in order to remove carbonic acid, transferred to the flask E, and the portion adhering removed by rinsing the measure with a little water. The beer is then distilled until the whole of the alcohol has passed over, the distillate made up with water to the volume of 1500 grain measures at 60° F., and its density observed. The residue in the flask E is made up with water to the volume of 1500 grain measures at 60° F., and its density observed. Then from these two data the original gravity is ascertained as above directed.

For estimating the amount of acid, 1000 grain measures of the beer is mixed with a solution of ammonia (sp. gr. 0·9986) until neutralized. The number of grain measures required indicates the per-centage of acetic acid.

Another method of examining beer consists in estimating the amount of water, by observing the quantity of chloride of sodium dissolved by a known quantity of the beer, before and after removing the alcohol, and is based upon the following data:—

1. The solubility of chloride of sodium is constant for a range of temperature from 50° to 100° F.;—36 parts of the salt dissolving in 100 parts of water, and the saturated solution contains 2·7778 parts water for 1 part of the salt.

2. The presence of alcohol and extract does not augment the solvent action of water upon chloride of sodium; so that the amount of this salt dissolved by beer will be inversely proportionate to the amount of these constituents; thus, if 1000 grains of the beer dissolves 312·7 grains chloride of sodium, the amount of water would be 868·61 grains, for—

$$36 : 100 = 312{\cdot}7 : 868{\cdot}61,$$

and the remainder, 131·39 grains, would be the sum of the other ingredients of the beer, alcohol, extract, and carbonic acid. The amount of extract is estimated in the same manner, after separating the alcohol, and the amount of alcohol is found from the difference between the two results.

The apparatus, fig. 73, used for this experiment is called a "hallimeter," and consists of a tube 5 inches long, an inch and a half wide at the upper end, and a quarter of an inch

wide at the lower closed end, which is graduated so that each division represents the volume of one grain of chloride of sodium powdered, and passed through a sieve with meshes 0·0673 line by 0·0757 line, and wires 0·0458 line thick.

Fig. 73.

Experiment.—For estimating the sum of alcohol and extract, 1000 grains of the beer is weighed in a glass flask, 330 grains of powdered chloride of sodium introduced, the flask immersed for ten minutes in water at 100° F., and frequently shaken meanwhile. By this means the solution of the salt is facilitated and the carbonic acid is expelled. The flask is cooled, dried, and weighed; the loss of weight represents carbonic acid. Then by closing the mouth of the flask with the thumb and reversing it, the salt remaining undissolved may be collected in the neck, transferred, together with the liquid, to the hallimeter tube, and its deposition in the narrow portion of the tube facilitated by stirring with a wire and by shaking the tube. The volume of the salt is then read off, the quantity deducted from 330 grains, and the quantity, x, of water corresponding to the quantity, q, of salt dissolved, calculated by the proportion,—

$$x : q = 100 : 36.$$

The total amount of alcohol and extract is found by deducting this quantity of water from that of the beer operated upon.

If, for instance, the quantity of salt remaining undissolved is 17·3 grains, the quantity of water corresponding to the 312·7 grains dissolved will be 868·61; for,—

$$36 : 100 = 312{\cdot}7 : 868{\cdot}61.$$

Consequently the beer contains in 1000 parts 868·61 parts of water, and the remainder, 131·39 grains, is the sum of the carbonic acid, alcohol, and extract.

It then remains to estimate the amount of extract alone, and for this purpose 1000 grains of the beer is evaporated

to rather less than one-half, in a similar flask, so as to remove the whole of the alcohol and carbonic acid, water added until the residue weighs 500 grains, then 180 grains of salt, and the operation repeated as before. The quantity, q', of salt dissolved is found by deducting the residue from 180, the corresponding quantity, x', of water calculated as in the former experiment, and when deducted from the weight of the liquid gives the quantity of extract as residue.

If, for instance, the quantity of salt remaining undissolved is 21·3 grains, the quantity of water corresponding to the 158·7 grains dissolved will be 440·83 grains; for,—

$$36 : 100 = 158{\cdot}7 : 440{\cdot}83.$$

Consequently the liquid would contain in 500 parts 440·83 parts water, and the remainder, 59·17 grains, would be the amount of extract in 1000 parts of beer.

Then by adding to this amount, that of the carbonic acid estimated in the first instance and deducting the sum of both constituents from the total amount of carbonic acid, extract, and alcohol previously estimated, the amount of alcohol is found as remainder. Supposing the amount of carbonic acid in the previous instance to be 1·5, the amount of alcohol would be 70·72, for—

$$59{\cdot}17 + 1{\cdot}5 = 60{\cdot}67, \text{ and } 131{\cdot}39 - 60{\cdot}67 = 70{\cdot}72.$$

When the proportions of beer and salt used are those indicated above, the following table obviates the necessity of calculating in each instance the total amount of extract and alcohol, or the amount of extract corresponding to the quantity of salt left undissolved. For the first experiment, the number of grains of salt undissolved is found in the first column, and opposite to it in the second column is the corresponding amount of alcohol and extract conjointly. In the second experiment, the number of grains of salt left undissolved is found in the first column, and opposite to it in the third column is the corresponding amount of extract. When the quantity of salt in either case contains a fraction, the corresponding amounts of alcohol and extract, or of extract alone, are sought in the small table below and added to the amounts already obtained for the whole numbers.

Residue of salt in grains.	Corresponding amount of alcohol and extract.	Corresponding amount of extract.	Residue of salt in grains.	Corresponding amount of alcohol and extract.	Corresponding amount of extract.
0	83	—	21	142	58
1	86	—	22	144	61
2	87	—	23	147	64
3	92	—	24	150	67
4	94	—	25	153	69
5	97	—	26	156	72
6	100	—	27	158	75
7	103	—	28	161	78
8	106	22	29	164	81
9	108	25	30	167	83
10	111	28	31	169	86
11	114	31	32	172	89
12	117	33	33	175	—
13	119	36	34	178	—
14	122	39	35	181	—
15	125	42	36	183	—
16	128	44	37	186	—
17	131	47	38	189	—
18	133	50	39	192	—
19	136	53	40	194	—
20	139	56			

Residue of salt in fractions of grains.	Corresponding amount of alcohol and extract.	Corresponding amount of extract.	Residue of salt in fractions of grains.	Corresponding amount of alcohol and extract.	Corresponding amount of extract.
0·1	0	0·4	1	0·7	2
2	1	5	2	8	2
3	1	6	2	9	3

An important correction is still to be made in order to ascertain the amount of absolute alcohol. This is owing to the fact that the quantity of water estimated in the first experiment from the quantity of salt dissolved, does not represent the whole of the water in the beer, a certain portion always remaining combined with the alcohol. In the second experiment this is not the case, and the calculated result represents the true amount of water.

The quantity of water thus retained by the alcohol is proportionate to the amount of alcohol in the beer, consequently it is requisite to have a table constructed so as to show the

amount of absolute alcohol and water in any quantity of alcohol indicated in the first experiment. This table has been constructed from experimental data obtained with liquids containing known proportions of absolute alcohol.

Quantity of alcohol found.	Amount of absolute alcohol by volume.	Differences.	Quantity of alcohol found.	Amount of absolute alcohol by volume.	Differences.
39	21·496		79	43·899	0·519
40	22·052	0·556	80	44·347	0·519
41	22·608	0·556	81	44·847	0·525
42	23·165	0·557	82	45·366	0·519
43	23·721	0·556	83	45·885	0·519
44	24·277	0·556	84	46·404	0·519
45	24·833	0·556	85	46·923	0·519
46	25·389	0·556	86	47·442	0·519
47	25·946	0·557	87	47·961	0·519
48	26·502	0·556	88	48·480	0·519
49	27·058	0·556	89	48·999	0·519
50	27·615	0·557	90	49·518	0·519
51	28·171	0·556	91	50·037	0·519
52	28·727	0·556	92	50·556	0·519
53	29·284	0·557	93	51·075	0·519
54	29·840	0·556	94	51·504	0·519
55	30·396	0·556	95	52·113	0·519
56	30·952	0·556	96	52·632	0·519
57	31·561	0·609	97	53·151	0·519
58	32·170	0·609	98	53·670	0·519
59	32·779	0·609	99	54·187	0·517
60	33·388	0·609	100	54·702	0·515
61	33·996	0·608	101	55·217	0·515
62	34·605	0·609	102	55·732	0·515
63	35·214	0·609	103	56·247	0·515
64	35·823	0·609	104	56·762	0·515
65	36·432	0·609	105	57·277	0·515
66	37·041	0·609	106	57·792	0·515
67	37·597	0·556	107	58·307	0·515
68	38·102	0·505	108	58·821	0·515
69	38·620	0·518	109	59·373	0·515
70	39·138	0·518	110	59·852	0·515
71	39·656	0·518	111	60·368	0·516
72	40·175	0·519	112	60·883	0·515
73	40·694	0·519	113	61·398	0·515
74	41·213	0·519	114	61·913	0·515
75	41·732	0·519	115	62·428	0·515
76	42·251	0·519	116	62·943	0·515
77	42·770	0·519	117	63·458	0·515
78	43·289	0·519	118	63·973	0·515

TABLE OF ALCOHOL PROPORTIONS (*continued*).

Quantity of alcohol found.	Amount of absolute alcohol by weight.	Differences.	Quantity of alcohol found.	Amount of absolute alcohol by volume.	Differences.
119	64·488	0·515	135	72·790	0·504
120	65·004	0·516	136	73·295	0·504
121	65·519	0·515	137	73·799	0·504
122	66·034	0·515	138	74·303	0·504
123	66·549	0·515	139	74·808	0·504
124	67·064	0·515	140	75·312	0·504
125	67·579	0·516	141	75·817	0·504
126	68·095	0·516	142	76·322	0·504
127	68·610	0·515	143	76·826	0·504
128	69·125	0·515	144	77·330	0·504
129	69·636	0·511	145	77·835	0·504
130	70·134	0·058	146	78·339	0·504
131	70·642	0·508	147	78·844	0·504
132	71·277	0·504	148	79·348	0·504
133	71·781	0·504	149	79·853	0·504
134	72·286	0·505	150	80·357	0·504

Thus the quantity of alcohol, 70·72, indicated by experiment, in the above instance would be found by reference to the table to contain 39·51 parts by measure of absolute alcohol, and 31·21 parts of water, which may be added to the quantity of water indicated by the salt dissolved.

The following tables show the average amounts of alcohol in various kinds of beer, wine, spirit, &c.

	Per-centage by weight of		
	Absolute alcohol.	Extract.	Carbonic acid.
Ale, Barclay's	6·90	6·02	0·15
„ London	8·08	15·88	
„ Burton	6·62	14·97	0·04
Porter, London	4·76	7·53	
	6·91	6·80	
„ Barclay's XX	6·10	5·98	0·18
Belgian beer	4·20	3·84	
Bavarian „	4·94	13·03	0·77
„ Bock-beer	4·10	7·61	0·19
	4·70	7·48	0·18
Darmstadt beer (for storing) about	3·00	4·00	0·18
Bohemian „ (draught) „	2·50	4·50	0·5

	Per-centage amount of	
	Proof-spirit by measure.	Absolute alcohol by measure.
e	26·195	21·75
.....	23·886	22·61
.....	22·113	18·37
t, Claret	7·162	6·18
	18·199	15·11
a	20·184	16·76
.....	21·121	17·45
.....	19·247	15·98
.....	20·619	17·22
leira	20·519	17·04
.....	18·288	15·91
.....	17·017	14·31
me.....	5·794	5·00
	12·601	10·46
y	7·186	6·19
ermitage	15·459	13·34
	19·436	16·14
.....	13·739	11·40
.....	9·271	8·00
	15·424	13·31
trave.....	14·274	11·84
ac	19·838	11·84
i	13·683	11·36
n	19·225	15·96

	Per-centage amount of	
	Proof-spirit by measure.	Absolute alcohol by measure.
Cape Madeira.....	20·195	16·77
Muscat	20·351	17·00
Constantia	22·024	18·29
Tinto	14·831	12·32
Schiratz	17·307	14·35
Syracuse	17·039	14·15
Nice	16·315	13·64
Tokay	11·018	9·15
Raisin wine.....	28·737	23·86
Drained grape wine	20·195	16·77
Lachrymæ Christi	21·968	18·24
Currant wine	22·916	19·03
Gooseberry wine...	13·203	10·96
Elder wine	11·006	9·14
Cyder		
Perry		
Brown Stout	7·583	6·30
Ale	9·902	8·00
Porter	4·684	3·89
Rum	59·861	49·71
Hollands.....	57·542	47·77
Scotch Whiskey...	60·575	50·20
Irish do.	60·107	49·91

CHAPTER XIV.

SUGAR, STARCH, FLOUR, BREAD, MILK, BUTTER, TEA, COFFEE, CHOCOLATE, ISINGLASS, ETC.; THE TESTS OF THEIR PURITY AND THE METHODS OF VALUATION.

THE substances treated of in this chapter are mostly important either as articles of food, or as constituents of food. Some of them are also used for other purposes in the arts. The methods of valuation applicable to them depend in great measure upon the purposes for which they are employed.

Sugar. The varieties of sugar that are of any great importance in commerce, &c., are cane-sugar, $C^{12}H^{11}O^{11}$, grape-sugar, $C^{12}H^{14}O^{14}$, and starch-sugar, $C^{12}H^{14}O^{14}$, together with the uncrystallizable sugar, $C^{12}H^{9}O^{9}$, and fruit-sugar, $C^{12}H^{12}O^{12}$, usually associated with the two former kinds. Other kinds of sugar, such as milk-sugar, mannite, &c., are less important.

The most frequent and probably the most important kind of sugar is that obtained from sugar-cane, *Saccharum officinarum*; beet-root, *Beta vulgaris*, and more rarely from the stalks of maize, *Zea mais*, or the sap of the maple, *Acer saccharinum*, &c. The sugar originating from all these sources is chemically the same, and for all practical purposes there is so little difference between the products obtained from them, that there is no great probability of any question arising as to what may be the plant from which any particular sample of sugar was obtained. On the other hand, it is more frequently desirable to know whether grape-sugar is mixed with cane-sugar, and whether grape-sugar syrup is not mixed with honey or cane-sugar syrup.

Estimation of sugar.

The amount of sugar—either cane-sugar or grape-sugar—in any substance may be estimated from the quantity of car-

bonic acid produced by the fermentation of the sugar with yeast. For this purpose a known quantity of the substance is dissolved in water, the solution introduced into the flask, *a*, fig. 33, and mixed with yeast. The flask, *b*, containing sulphuric acid, is then attached as in the alkalimetric operation (p. 66), and the apparatus kept for some days at a temperature of 77° F. until the evolution of gas has ceased. The loss of weight then gives the quantity, *q*, of carbonic acid, and the quantity, *x*, or *x'*, of sugar is calculated thence by one of the proportions:

$$x : q = C^{12}H^{11}O^{11} : 4CO^2, \text{ or } = 171 : 88,$$

$$x' : q = C^{12}H^{14}O^{14} : 4CO^2, \text{ or } = 198 : 88,$$

according as the sugar to be estimated is cane- or grape-sugar.

If it is desirable, the result thus obtained may be checked by estimating the quantity of alcohol, produced at the same time, by distilling the contents of the flask *a*, and proceeding as directed at p. 332, for the estimation of alcohol.

Cane-sugar may also be estimated, even when associated with grape-sugar, by a method based upon the following data:

1. Cane-sugar combines with lime in definite proportions, forming a substance that has the alkalinity of lime, and is not decomposed at 212° F.

2. The corresponding compound of grape-sugar and lime is decomposed when its solution is heated to 200° F. with production of a substance that is perfectly neutral.

3. Water dissolves only 0·1 per cent. of lime, while sugar solution dissolves considerably more, owing to the production of the lime-sugar compound, $2C^{12}H^{11}O^{11}, 3CaO$, which is soluble in water.

The experiment is made with 10 grms. of the sugar, which is intimately mixed with about an equal weight of slaked lime and a few drops of water, the mass transferred to a filter, and the filtrate passed through the lime again so as to saturate the whole of the sugar, the residue washed with water, and the liquid diluted with water to the volume of one litre. A tenth of this solution is then diluted with twice or three times its volume of water mixed with litmus, and the alkalimetric experiment made with 20 cubic centimeters of the normal sulphuric acid solution (p. 56) diluted to 100 cubic centimeters. The number of centimeters of test-solution

requisite to neutralize the lime will express the per-centage amount of sugar, after deducting 2 cubic centimeters as a correction for the lime dissolved by the water.

When cane-sugar is associated with grape-sugar, the liquid that has been saturated with lime is boiled previous to the alkalimetric experiment; by this means the compound of grape-sugar with lime is decomposed, and the lime combined with cane-sugar, alone remains recognizable by the test acid.

In examining saccharine liquids such multiples of 10 cubic centimeters as correspond with the amounts of sugar they contain are measured and treated in the same manner as the sugar solution.

If it is desirable to estimate the total amount of sugar when the two kinds are associated together, two experiments must be made, in one of which the quantity of lime is estimated before boiling the liquid, and the difference between the two results thus obtained indicates the amount of grape-sugar.

Another method of estimating sugar is based upon the following data:

1. Grape-sugar precipitates red suboxide of copper from alkaline solutions containing oxide of copper at 140° F.

2. Cane-sugar does not exercise this reducing action under the same conditions.

3. Cane-sugar may be easily converted into grape-sugar.

The reaction that takes place between grape-sugar and oxide of copper under these circumstances is of such a nature that 180 parts of grape-sugar effect the reduction of 10 equivalents of oxide of copper.

The test-solution is made by dissolving 140 grms. of neutral tartrate of potash in a small quantity of water, mixing it with from 600 to 700 grms. of caustic soda solution (1·12 sp. gr.), then adding a solution of 36·46 grms. sulphate of copper in water, filtering and diluting with water to the volume of one litre. A dark blue liquid is thus obtained which may be kept for some time, in the dark, without decomposition, and is not altered by boiling. Its volumetric value is estimated by direct experiment. For this purpose 1 grm. of pure crystallized cane-sugar is dissolved in water, and converted into grape-sugar by boiling the liquid for half an hour with a few drops of sulphuric acid.

This solution is diluted with water to the volume of 100 cubic centimeters; consequently each cubic centimeter contains 0·0116 grm. grape-sugar and represents 0·01 grm. of cane-sugar. Then 50 cubic centimeters of the copper solution is placed in a capacious porcelain dish, mixed with some strong caustic potash solution, heated to the boiling-point, and the sugar solution gradually added from a burette, until the liquid becomes colourless, and the whole of the copper is precipitated as red suboxide.

The best way of ascertaining whether the whole of the copper is precipitated, is to test a drop of the liquid from time to time towards the end of the operation with ferrocyanide of potassium solution, feebly acidified, which produces a reddish-brown precipitate or coloration so long as a trace of copper remains in solution.

The number of cubic centimeters of sugar solution requisite to precipitate the whole of the copper gives the quantity of cane-sugar which, when converted into grape-sugar, is equivalent to 50 cubic centimeters of the copper solution, which is diluted, if necessary, so that 100 cubic centimeters represent exactly 0·5 grm. of cane-sugar.

In order to examine a mixture of cane-sugar and grape-sugar by this method two solutions are made, each containing 10 grms. of the sugar; one of the solutions is boiled for half an hour with sulphuric acid, for the purpose of converting the whole of the cane-sugar into grape-sugar, and then both solutions are diluted with water to the volume of a litre. An experiment is then made as above directed with each of these solutions, 50 cubic centimeters of the copper solution being used in each, and the volume of sugar solution requisite for precipitating the copper in each indicates the amount of grape-sugar present, the difference between the two results giving the quantity of grape-sugar equivalent to the cane-sugar present.

Thus, for example, if 100 cubic centimeters of the sugar solution that has not been boiled with acid, representing 1·0 grm. of the sugar under examination, exactly precipitates the copper from 50 cubic centimeters of the test-solution, that quantity will contain as much grape-sugar as is equivalent to 0·250 grm. cane-sugar, or 0·289 grm., and the amount of grape-sugar in the sample examined will be 28·95 per cent.

If, in the experiment with the sugar solution that has been boiled with acid, 30 cubic centimeters precipitate the copper from 50 cubic centimeters of the copper solution, this quantity will contain as much grape-sugar as is equivalent to 0·250 grm. cane-sugar, or 0·289 grm., and the total amount of grape-sugar would be 83·33 per cent.

Then the difference between the two results will give the amount of grape-sugar equivalent to the cane-sugar converted by boiling with acid; thus:

Total amount of grape-sugar	96·49			
Actual per-centage of grape-sugar	28·95		28·95	grape-sugar.
Amount of grape-sugar equivalent to cane-sugar present	67·54	=	58·33	cane-sugar.
			12·72	impurities.
			100·00	

For 67·54 : 58·33 = 198 : 171.

The amount of crystallizable cane-sugar may also be estimated by washing a known quantity of the sample with a saturated solution of sugar, which removes only the syrup and uncrystallizable sugar, &c. For this purpose 10 grms. of the finely powdered sugar, mixed with 4 cubic centimeters of alcohol, is shaken with 10 cubic centimeters of a liquid consisting of 800 cubic centimeters alcohol, 40 cubic centimeters acetic acid, and 40 grms. of sugar; the coloured liquid is poured off, and the operation repeated, if requisite, with a fresh quantity of the liquid. When the sugar appears sufficiently washed, it is transferred to a weighed filter, the funnel covered with a glass plate, and when the liquid has ceased to drop through the filter, the sugar is weighed, dried at 212° F., and again weighed. The difference between the two weighings gives the quantity of liquid evaporated from the sugar solution, and as the amount of sugar in that solution is known, the corresponding quantity of sugar may be calculated and deducted from the weight of the filter and its contents.

The solution used for this experiment should be kept saturated by hanging in it a bag of sugar crystals.

The raw sugar met with in commerce is never pure, but contains admixtures of moisture, colouring, gummy and albuminous substances, fragments of cellular tissue, sand

and earthy particles, soluble salts, uncrystallizable sugar, certain organic acids, &c. Some of the admixtures are in no other way detrimental than in reducing the value of the sugar according to their amount; others are, in addition to this, prejudical in the refining operation, and determine the conversion of a considerable amount of sugar into molasses.

Water is indicated to some extent by the appearance of the sugar; its amount may be estimated by heating a weighed quantity of the sugar to 230° F. until the weight remains constant.

Sand, &c. are indicated by a residue insoluble in water, and may be estimated by weighing the washed and dried residue obtained from a known quantity of sugar.

Gummy and albuminous substances are indicated, on the addition of basic acetate of lead solution, by a precipitate, and may be estimated by weighing the washed and dried precipitate obtained from a known quantity of sugar, and then igniting the precipitate to ascertain the amount of organic substances it contains.

Mineral salts are indicated by the usual tests in the residue obtained by heating 5 or 10 grms. of the sugar upon a clean plate in a muffle, until all the carbon is burnt.

Glucose or starch-sugar, indicated, when a solution of the sugar is boiled with caustic soda, by a brown coloration; or when a concentrated solution of the sugar is mixed with hydrate of potash, heated to 212° F., diluted with water, and mixed with a few drops of nitrate of cobalt solution. In the absence of starch-sugar, a blue precipitate is produced directly, and it becomes green after some time, but when starch-sugar is present, this reaction is not produced. Concentrated solutions of starch-sugar give a dirty brown precipitate.

Starch-sugar is also indicated when a solution of the sugar is heated to 140° F. with an alkaline solution of oxide of copper, by a red precipitate of suboxide of copper. Cane-sugar solution does not give this reaction, until after it has been boiled some considerable time with the copper solution.

Honey is the only kind of grape-sugar that is met with in commerce. It contains, besides crystallizable grape-sugar, glucose or uncrystallizable fruit-sugar.

The substances with which it is most frequently adulterated, are,—

Starch-sugar; indicated probably, when the honey is boiled with six or eight times its volume of alcohol (80 per cent.), by an insoluble residue which, when dried, presents the appearance of gum. Such a residue may consist wholly or in part of gelatin, so that it must be tested to ascertain whether this is the case. For this purpose the test by charring with lime gives the quickest indication, and a solution of tannic acid, mixed with the water solution of the residue, gives a precipitate with gelatin but not with dextrin. However, no means are known by which starch-sugar can be detected or estimated with certainty in honey.

Starch or flour; indicated, when the honey is mixed with cold water, by a residue, which gives with solution of iodine the blue colour characteristic of starch, see p. 358. The amount may be estimated by collecting and weighing the residue.

Gelatin; indicated, when the honey is dried and heated in a test-tube with caustic lime, by the evolution of ammonia, which gives a blue colour to litmus paper held over the mouth of the tube.

Fig. 74.

Starch; an almost universal constituent of certain parts of plants.

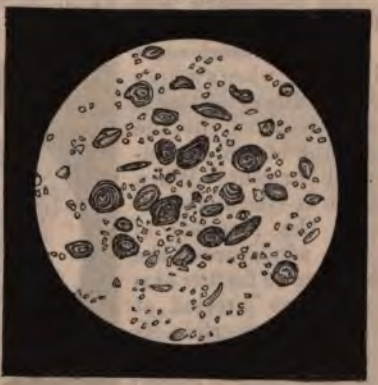
This substance consists of small granules with a cellular structure varying in size and appearance according to the plant from which it is derived. The general characteristics of starch-granules are best illustrated by the starch of the potato, which, when examined by the aid of the microscope, presents the appearance shown in the accompanying woodcut (fig. 74). In most kinds of starch-granules there is a point, *a*, round which the substance of the granules is arranged in regular layers represented by concentric lines.

This point is called the hilum, and its appearance is more or less characteristic; in some kinds of starch the hilum is not so marked as in others, and as a general rule it is more apparent when the granules are dry. In the accompanying drawings of starch-granules, those marked *a* are represented as seen when dry, and those marked *b* are represented as seen when moist.

Fig. 75.

The symmetric structure, and the position of the hilum, are rendered still more apparent when the granules are examined by the aid of polarized light, as shown by fig. 75.

Fig. 76.



The starch of commerce is generally the produce of varieties of *Triticum*, of *Oryza sativa*, and of *Solanum tuberosum*. The starch of other plants also constitutes, under various names, articles of commerce.

The granules of wheat-starch (fig. 76) appear by aid of the microscope to vary in size; the smaller being nearly spheroidal, the larger ones lenticular. The hilum is round or elongated and surrounded by concentric rings.

The starch-granules of barley, oats, and rye much resemble those of wheat; those of barley-starch (fig. 77) are somewhat larger, their shape less regular, and the hilum and rings are

scarcely perceptible;—those of oats (fig. 78) are for the most part more angular, the hilum is tolerably distinct, but no rings are perceptible, and crosses do not appear when they are seen by polarized light;—those of rye (fig. 79) are for the most part the largest of the four with fewer small granules, their shape is irregularly lenticular, rarely angular; the hilum is very marked, and generally radiate.

Fig. 77.

The starch-granules of rice (fig. 80) are very small, polygonal, decidedly angular, and without visible hilum or concentric rings.

The substances with which starch is likely to be adulterated, are,—

Fig. 78.

Mineral substances; indicated, when a weighed quantity of the starch is incinerated, by the amount of ash exceeding 1 per cent. When there is reason to suspect the presence of such admixtures, the ash may be further examined, as directed at p. 219, to ascertain its nature.

Other kinds of starch; indicated, when the starch is examined by the aid of the microscope, by the appearance of the granules.

Potato-starch mixed with wheat-starch is indicated, when the starch is gently rubbed with water, and the filtered

liquid mixed with iodine solution, by the production of a blue colour. Wheat-starch treated in this way gives a liquid that becomes only yellow or reddish-yellow. It is necessary, however, to be careful in rubbing the starch, not to exercise too much force, so as to break the granules of wheat-starch, since in that case a blue colour would be produced even in the absence of potato-starch.

Fig. 79.

Potato-starch may be distinguished from wheat-starch also by the effects produced upon them by an alkaline solution containing 1·75 per cent. caustic potash. Wheat-starch is scarcely altered in appearance when mixed with thirty times its weight of this solution; the liquid remains thin and the starch-granules sink to the bottom when the liquid is diluted.—Potato-starch gives under the same circumstances a thick opaline jelly within a minute of mixing; the granules swell considerably, and when more liquid is added appear as if completely dissolved. A mixture of equal parts wheat-starch and potato-starch gives a jelly within two minutes. A mixture of four parts wheat-starch and one part potato-starch gives a thick opake milky mucilage. A mixture containing one-tenth potato-starch gives

Fig. 80.

an opake milky mucilage, which does not run in drops like the liquid obtained with pure wheat-starch.

The most distinctive character of starch is the production of an intense blue colour when iodine is mixed with its solution in water.

Flour; the ground seeds of certain grasses and leguminous plants.

The various kinds of flour contain as normal ingredients starch, albuminoid substances, dextrin, sugar, cellulose, water, fat, and a certain amount of mineral substance.

The various kinds of flour differ considerably, not only according to the grain from which they are prepared, but also according to the quality, age, or condition of the grain, the mode of grinding, and the sifting or dressing it undergoes.

The quality of flour and the characters it presents will therefore be materially influenced by one or other of these circumstances, and, as a general rule, the determination of these particulars calls for greater attention, in the examination of flour, than the search for admixtures, either accidental or fraudulent.

The substances with which flour is likely to be adulterated, are,—

Mineral substances; indicated, when a weighed portion of the flour is incinerated, by the amount of the ash exceeding 2 per cent.

Other kinds of flour; this is probably the most frequent adulteration. Although examination of the starch-granules by the aid of the microscope may serve to distinguish between wheat-flour and that of other grain, it is questionable whether the presence of other kinds of flour mixed with wheat-flour can be detected with certainty by the use of the microscope even when the amount is considerable, on account of the great similarity of the starch-granules from different plants, the diversity of those from the same plant, and also the small number of granules that can be brought within the microscopic field.

Potato-starch or flour may be detected by means of a weak solution of caustic potash (p. 357).

Besides barley, oat, rye and rice-meal, wheat-flour may be adulterated with the meal of maize or leguminous plants.

The starch-granules of maize (fig. 81) are rounded, more or less irregular, and sometimes mullar-shaped; the hilum is either circular or radiate, and very distinct: no rings are perceptible.

Fig. 81.

The starch-granules of peas (fig. 82) are long, irregularly shaped and more uniform in size than those of wheat; the hilum presents the appearance of a longitudinal depression or slit, sometimes with lateral or transverse branches.

The presence of meal of leguminous plants is also indicated by the deliquescent character of the ash, by its alkaline reaction upon turmeric paper, and when its aqueous solution is mixed with nitrate of silver solution, by the production of a white precipitate which is darkened by exposure to light.—The ash of pure wheat-flour is granular, sandy, the aqueous solution does not affect turmeric paper, and the precipitate produced with nitrate of silver does not darken by exposure to light.

Fig. 82.

In the quantitative examination of flour, the several data to be ascertained are the amounts of—

Moisture; estimated by drying a known quantity of the flour at 212° F., and weighing again until the weight remains constant.

Fat; estimated by digesting a weighed quantity of the dried flour with successive quantities of ether; evaporating the clear liquid and weighing the residue, dried at 212° F.

Starch; estimated from the quantity of grape-sugar it yields. For this purpose the residue, left after separating fat and dextrine, is boiled with a small quantity of sulphuric acid, until iodine no longer produces a blue colour in the liquid, the sulphuric acid separated by agitating the liquid with carbonate of baryta, and filtering; the filtrate is then evaporated to dryness at 212° F., the residue of grape-sugar weighed, and the corresponding amount of starch ascertained by calculation, 18 parts of grape-sugar representing 16 parts of starch.

Dextrine; estimated by digesting a known quantity of the flour with water at the ordinary temperature, evaporating the liquid to dryness at 212° F., and weighing the residue.

Gluten, albumen, &c.; estimated from the quantity of nitrogen obtained in the state of chloride of ammonium and platinum, as directed at p. 197; 16 parts of nitrogen representing 100 parts of dry albuminous substance.

The amount of gluten in wheat flour may be estimated approximatively by kneading the flour with water and washing the dough upon a cloth in a current of water, by which means the starch is removed and the greater part of the gluten remains adhering to the cloth.

Bran; estimated by warming a known quantity of the flour with water upon a steam-bath, straining the liquid through fine muslin, washing the residue until the water passes through clear, then drying and weighing it. This dry residue represents twice its weight of bran in the natural state.

Cellular membranes, lignin; estimated by covering a known quantity of flour with sulphuric acid diluted with rather more than its own weight of water, digesting the mixture for twenty-four hours, heating it on a steam-bath until the acid liquid no longer becomes turbid on the addition of water; it is then diluted with water, filtered, the contents of the filter washed with warm water, dried at 212° F., and weighed.

Mineral substances; estimated as ash, by igniting a known quantity of the flour in a shallow platinum capsule until the whole of the carbon is burnt, and weighing the residue.

The ash thus obtained will contain, besides the mineral constituents of the corn, a certain quantity of substance originating from the grinding stones, but this should not amount to more than half an ounce in the hundredweight of flour, an amount much less than that of the mineral constituents of various kinds of flour separated from bran, which in wheat- and rye-flour is about 1 per cent., in barley-flour 1·5 per cent., and in oat-flour 2·0 per cent., so that when any kind of flour is found to contain more than 2 per cent. of mineral substances, it may be safely inferred that they have been mixed with the flour either accidentally or fraudulently.

If it is desirable, the ash may be examined, as directed in Chapter IX., to ascertain the substances it contains, in case there is reason for suspecting adulteration with mineral substances.

Bread is liable to adulteration with the same materials as flour; it may also contain—

Alum; indicated by the presence of alumina in the ash.

Sulphate of copper; indicated by the presence of copper in the ash.

Both substances are tested for as directed in Chapter X.

Milk; an animal secretion; chiefly the produce of *Bos Taurus*.

It is essentially a water solution of casein, of a peculiar kind of sugar and certain saline substances, in which are suspended small globules of fat that communicate to the liquid its opacity. The amount of the several ingredients of milk varies according to the species, age, food and mode of life of the animal yielding it.

The density of normal cow's milk may vary between 1·026 and 1·032, but as this character is influenced in opposite ways by the amount of butter and by that of casein and sugar, it does not afford any precise indication of the goodness of milk.

The substances with which milk is most likely to be adulterated, are,—

Water; probably indicated, when a drop of the milk is placed on the finger nail, by the thinness and flattening of the drop; while in the case of good milk the drop retains its

convex form. This test is not very trustworthy, especially in its negative indications.

Dextrin, starch or *flour*; indicated, when the milk is mixed with a drop of iodine solution, by the production of a deep blue or brown colour.

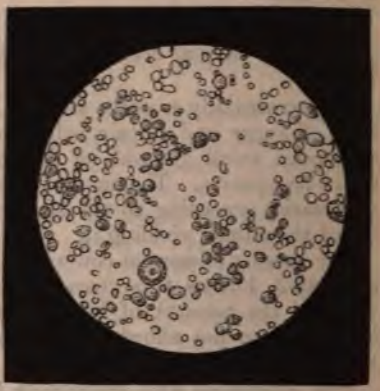
Brains of sheep, calves, &c.; indicated by the greyish colour of the milk, and, when it is left for a time undisturbed, by the deposition of a white sediment, containing fine threads of the cellular substance of the brain. Microscopic examination would be the best means of detecting the presence of brain.

Normal milk seen by the aid of the microscope presents the appearance represented by the drawing (fig. 83).

Chalk or *carbonate of magnesia*; indicated by a sediment which effervesces with acids.

Alkaline carbonates; indicated, when the heated milk is mixed with acetic acid, by the curdling not taking place immediately. The presence of alkaline carbonates is also indicated, when the milk is mixed with acetic acid, heated, the curd separated by filtration and washed, the filtrate then evaporated nearly to dryness, the residue mixed with absolute alcohol, the clear liquid evaporated to dryness, the residue ignited, and the ash weighed. This should not amount to more than 0·35 per cent.

Fig. 83.



Metallic substances; originating from vessels in which the milk has been kept, would be tested for in the ash as directed in Chapter X.

For the purpose of estimating the goodness of milk that does not appear to be adulterated, the best criterion is probably the amount of dry residue left after evaporation to dryness. This is estimated by mixing with a known quantity

of the milk an equal weight of powdered gypsum, and evaporating until it becomes pasty, and then drying the residue upon a steam-bath until its weight remains constant. The difference between the weight of the residue and that of the milk and gypsum, gives the quantity of water in the milk and, by subtracting this from the quantity of milk taken, the quantity of dry substance.

The amount of dry substance should be at least 12 per cent., and may be taken as an indication that no great abstraction of cream or dilution with water has been practised; sometimes it is as much as 14 per cent.

The amount of fat may be estimated by repeatedly digesting the dry residue, from the last operation, with ether, until nothing more is extracted, then filtering the liquid, evaporating, and weighing the dry residue.

The normal amount of fat varies from 3·5 to 5 per cent.

The amount of sugar may be estimated by the method already described for estimating grape-sugar by means of an alkaline solution of copper. For this purpose the fat and casein must be separated previously, and this is effected by heating a known quantity of the milk with a few drops of acetic acid, separating the curd by filtration, and then proceeding as directed at p. 350.

The normal amount of sugar is 5·27 per cent.

The amount of cream that milk is capable of yielding may be estimated by leaving a known quantity at rest for some hours in a tall graduated tube, and then observing the volume of the cream collected at the surface.

Butter; the fat obtained from cow's milk.

The substances with which butter is likely to be adulterated, are,—

Water, *flour*, &c., and perhaps *earthy substances*; indicated, when a known quantity of the butter is melted with water, then allowed to solidify, and weighed, by the reduction of weight amounting to more than 5 per cent.

The presence of amylaceous substances would be indicated by means of iodine solution added to the liquid; earthy substances or casein would appear as a sediment in the liquid, and colouring substances would be indicated by the appearance of the liquid.

Tea; the dried leaves of *Thea bohea*.

The substances with which tea is adulterated, are,—

Prussian blue or *indigo*; indicated, when the tea is examined by the aid of the microscope, by the presence of a powder adhering to the surface; they may also be detected by shaking the tea with water, separating the leaves by filtering through muslin, then collecting the sediment, and examining it to ascertain whether it consists of prussian blue or indigo; the latter is less frequent.

Gypsum; indicated, when the tea is shaken with water, by the production of a white precipitate in the liquid, by chloride of barium.

Chromate of lead; tested for in the sediment obtained by shaking the tea with water, as directed in Chapter X.

Copper salts; tested for in a similar manner, either in the sediment or in the decoction.

Lie tea—a fictitious tea, made of tea dust, or ground rice, and gum,—indicated, when the tea is digested with water, by not unfolding like tea leaves, and when digested with hot water by falling to powder.

The amount of ash yielded by good tea is 5 per cent. or at the most 6 per cent.; lie tea yields from 37 to 45 per cent. ash.

Coffee; the dried berries of *Coffea arabica*.

The only adulteration of raw coffee is the colouring of yellow berries. This is effected with the same substances that are used for colouring tea, and they may be detected in a similar manner.

The substances with which roasted and ground coffee are adulterated, are,—

Chicory; indicated, when the suspected coffee is placed upon water in a test-tube, by the immediate brown coloration of the water. Pure coffee is not wetted at once and does not colour water until it has been in contact with it for some time. When chicory or a mixture of chicory with coffee is placed upon water, streaks of coloured liquid are seen to fall down through the water more or less rapidly, according to the amount of chicory. With some little practice the amount of chicory mixed with coffee may be ascertained with tolerable accuracy.

Roasted corn, *acorns*, and *leguminous seeds*; indicated, when

the decoction is mixed with iodine solution, by the production of a blue colour, which is not the case with a decoction of pure coffee.

Coffee grounds; indicated best by estimating the amount of extract yielded by the coffee. This is effected by boiling the coffee several times with water, filtering the liquid, evaporating to dryness upon a water-bath, and drying the residue. Good coffee ought to yield about 37 per cent.

A number of other substances more or less improbable are stated to be used for adulteration of coffee.

Chocolate; a preparation of the seeds of *Theobroma cacao*.

The substances with which chocolate is adulterated, are,—

Starch or *amylaceous substances*; indicated, when the filtered hot infusion of the suspected substances is mixed with iodine solution, by the production of the deep blue colour characteristic of starch.

Sugar can hardly be regarded as an adulteration of chocolate except when exceeding a certain amount.

Iron ochre and *other mineral substances*; indicated, when the chocolate is incinerated, by the usual tests applied to the ash.

The determination of the quality of chocolate is to be effected by estimating the amount of the several normal ingredients, and in the case of inferior kinds, of the admixtures present, by the methods already described for estimating sugar, fat, gum, starch, &c., but the results obtained will in all instances be somewhat indefinite as there is no absolute standard of quality with which they may be compared.

Gelatin; a nitrogenous constituent of bones and other parts of the animal organism.

The quantity of gelatin in the different states of glue, &c., may be ascertained by estimating the amount of water it is capable of absorbing, because the amount of water contained, either chemically or otherwise, in different kinds of gelatin, varies very much.

For this purpose a weighed quantity of the gelatin is immersed in cold water for twenty-four hours, the water that is not absorbed is then poured off, and the gelatin weighed. Very good kinds are thus increased in weight thirteen times, average kinds ten times, and inferior kinds six times.

The consistence of the residual jelly should also be observed; the more solid it is in proportion to the amount of water the better is the gelatin.

Isinglass; the dried coats of the sound or air-bladders, &c. of varieties of sturgeon (*Accipenser*), cod (*Gadus*), and other fish.

Isinglass is adulterated with

Gelatin, which is inserted between the leaves and rolled up with it.

The best indication of this adulteration is the amount of ash; isinglass yields only 0·9 per cent., while gelatin yields 4 per cent., and adulterated isinglass 1·5 per cent. or more.

CHAPTER XV.

TEXTILE MATERIALS; WOOLLEN CLOTH, SILK, COTTON, AND LINEN.

TEXTILE materials may be divided into two classes, according as they are derived from animals or plants. The most important of these are wool, silk, cotton, flax or linen, and hemp.

The structure and configuration of these fibres, as seen by means of the microscope, are their most characteristic features, and admit of their being distinguished from each other with greater certainty than is furnished by any chemical tests.

Silk; the produce of *Bombyx Mori*.

This fibre (fig. 84) has the most simple structure of any of those above mentioned. It consists of an indurated secretion, and by the aid of the microscope appears cylindric, smooth,

Fig. 84.

without organic structure or longitudinal cavity, presenting here and there at both sides a kind of narrow margin. The surface of the fibre is bright and the thickness uniform, although some fibres are thicker than others.

Wool; the produce of varieties of *Ovis*, &c.

This fibre (fig. 85) consists of a cylindrical aggregation of numerous cells, and appears by the aid of the microscope as a tube covered with epidermoid scales, which overlap each other like tiles, and are the chief distinctive characteristic of this fibre.

Fig. 85.

Linen; the produce of *Linum usitatissimum*.

This fibre (fig. 86) consists of longitudinal bast-cells, and appears by the aid of the microscope as a thin, round thread with a narrow longitudinal cavity; the surface is generally smooth and the fibre tapers off towards the end, and terminates in a blunt point. The diameter is not uniform, but greater at some places where the fibre appears flattened.

Fig. 86.

Cotton; the produce of *Gossypium herbaceum, religiosum, &c.*

This fibre (fig. 87) appears by the aid of the microscope as a flat ribbon-shaped cell, thicker at the edges than the middle, and twisted like a corkscrew, or irregularly curved. There is frequently a kind of reticular striation apparent on

the surface, and a broad longitudinal cavity more or less distinctly marked.

Fig. 87.

Hemp; the produce of *Cannabis sativa*.

This fibre (fig. 88) appears by the aid of the microscope of unequal thickness, terminating with a blunt point and frequently split at the ends; it has a tolerably wide longitudinal cavity, and the surface presents longitudinal striæ and pores appearing as transverse striæ.

Fig. 88.

Manilla or **New Zealand hemp**; the produce of *Phormium tenax*.

This fibre (fig. 89) appears by the aid of the microscope bright, colourless, cylindrical, terminating gradually in a

Fig. 89.

blunt point, with a longitudinal cavity, generally appearing like a simple line, and the surface destitute of any striation.

Chinese grass; the produce of *Corchorus capsularis*.

This fibre (fig. 90) appears by the aid of the microscope flat and ribbon-shaped, but not twisted like cotton, with a wide longitudinal cavity, and pores that frequently appear like transverse striæ.

Fig. 90.

The chemical tests by means of which the various fibres may be distinguished from each other, furnish results which are as a general rule less trustworthy than those obtained by the use of the microscope, especially when the fibres are woven together, but there is not much difficulty in distinguishing between animal and plant fibres by chemical means.

1. When the fibres are set fire to:

Animal fibres swell up, evolve vapour that has a peculiar odour, and turns litmus paper blue; leaving at the same time a bulky shining coal which is difficult to burn, and leaves a copious ash when burnt.	Plant fibres burn readily, evolve vapour that has an empyreumatic odour, and reddens litmus paper; leaving at the same time a coal that has the form of the fibre, and leaves little ash when burnt.

2. When the fibres are boiled with caustic soda solution (105 sp. gr.):

Animal fibres dissolve.	Plant fibres are little affected.

3. When the fibres are boiled for twenty minutes with solution of nitrate of mercury:

Animal fibres acquire a red colour.	Cotton and linen fibres remain colourless.

4. When the fibres are boiled with nitric acid:

Wool acquires a yellow colour, silk also, but in less degree.	Cotton and linen remain colourless.

5. When the fibres are immersed in a mixture of equal parts of sulphuric acid and nitric acid, for twenty minutes or less, and then washed with a large quantity of water:

Goat hair and silk are entirely dissolved. Wool becomes yellow or brown.	Plant fibres do not become coloured and their substance apparently little altered, but when dried, they burn with explosion.

6. When the fibres are moistened with a drop of chloride of tin, dried, and then heated:

Wool and silk do not appear altered.	Plant fibres soon become distinctly brown.

7. When white or bleached fibres are immersed in a solution of oxide of lead in caustic soda:

Wool and hair acquire a brown colour. Silk is not altered.	Plant fibres remain colourless.

8. When the fibres are moistened with solution of picric acid:

Wool and silk acquire a yellow colour.	Cotton and linen remain colourless.

9. When the fibres are burnt separately:

Linen fibre appears at the charred end, smooth and round.	Cotton fibre is splayed out at the end like a brush.

10. When a piece of the dry stuff, free from dressing, is immersed in concentrated sulphuric acid for half a minute or two minutes, according to the strength of the fabric, then washed with water, immersed in dilute solution of ammonia, so as to separate acid completely, and then dried:

Linen fibres remain unaltered or but little acted upon.	Cotton fibres are converted by the acid into a gummy substance which is dissolved by water.

A mixed fabric treated in this manner presents gaps which represent the position of the cotton fibres.

11. When a piece of the stuff is immersed in an alcoholic solution of madder for a quarter of an hour:

Linen fibres acquire a dirty reddish-yellow or orange-yellow colour.	Cotton fibres become bright yellow.

With an alcoholic tincture of cochineal:

Linen fibres become violet red.	Cotton fibres become bright red.

12. When a piece of the stuff, free from dressing, is immersed in olive oil:

Linen fibres become translucent like greased paper.	Cotton fibres remain opake and white.

A mixed fabric treated in this manner presents a striped appearance, which is more apparent by the aid of the microscope or a magnifying glass.

APPENDIX.

HYDROMETRY; TABLES OF DENSITY, ETC.

THE density, or the relative weight of equal volumes, of different substances is a character which sometimes affords indications of their chemical nature, and also of their value, particularly in the case of liquids; it is also frequently serviceable for other purposes, as in the analysis of water, soils, &c. The density, or as it is frequently called, the specific gravity of a substance is expressed numerically by comparison with water which is taken as unity.

The specific gravity of liquids is estimated by weighing in a narrow-necked flask (fig. 91) of known capacity for water at a given temperature. The point to which the flask is to be filled may be indicated by a scratch on the neck, or the flask may be fitted with a stopper made of thermometer tube, which, when inserted, admits of the escape of any excess over the proper volume.

Fig. 91.

The temperature at which the specific gravity of substances is to be estimated is generally 60° F. or 62° F., as prescribed by law, with an atmospheric pressure equal to 30 inches of mercury.

The flask used for the purpose should contain 1000, 500 or 250 grains of pure water, and should be exactly counterpoised by a weight in the opposite pan of the balance. The density of water being taken as unity, that of any other

liquid is found by dividing the weight, w, of a given volume of the liquid in question, by the weight, W, of an equal volume of water at the same temperature.

When the flask holds exactly 1000 grains of pure water, the specific gravity of any other liquid with which it may be filled is indicated at once by the weight of that volume of the liquid. But when the capacity of the flask is more or less than 1000 grains, the specific gravity, x, must be found by calculation, the requisite data being the weight, W, of water held by the flask and the weight, w, of an equal volume of the liquid in question.

$$W : w = 1000 : x.$$

The hydrometer is an instrument for ascertaining the density of liquids in accordance with the physical principle, that when a solid is immersed in a liquid its weight is to a certain extent supported or counterpoised. The extent to which this apparent reduction of weight takes place, depends upon the relative densities of the solid and liquid.

When the density of the solid is greater than that of the liquid in which it is suspended, the reduction of weight will be equal to the weight of a volume of the liquid that is equal to the entire volume of the solid.

When the density of the solid is less than that of the liquid, only a portion of the solid will be immersed, and the volume of this portion will be equal to a volume of liquid whose weight is equal to the entire weight of the solid. Consequently the greater the density of the liquid as compared with the solid, the smaller will be the volume of liquid displaced by the solid, and the reverse. Thus, for example, when a solid, having a volume of 2 cubic centimeters, and weighing 1 gramme, is immersed in a liquid having double the density, 1 cubic centimeter of which weighs 1 gramme, the solid will be half immersed in the liquid. If the liquid was four times as dense as water, only one-fourth of the solid would be immersed, and if the liquid was three-fourths as dense as water, two-thirds of the solid would be immersed; or, in other words, the portions of the solid immersed in liquids whose densities were in the relation of $2 : 1 : \frac{3}{4}$, would be in the relation of $\frac{1}{2} : \frac{1}{2} : \frac{2}{3}$, or inversely as the densities of the liquids.

The hydrometer (fig. 92) is constructed of glass or metal, and by making the upper part hollow, while the lower part is loaded with lead, such a relation is effected between the volume and the weight of the instrument, that it sinks to a depth, inversely proportionate to the density of the liquid in which it is immersed.

Fig. 92.

By making the instrument spindle-shaped, with a stem sufficiently long, and so adjusting the weight and volume that it floats upright in water, with about half the stem above the surface, the specific gravity of any liquid might be ascertained by means of it, but owing to the practical inconveniences that would attend the use of such an instrument, hydrometers are always specially adapted for liquids, either heavier or lighter than water, the zero or point to which the instrument sinks in water, being in one case at the bottom, in the other at the top of the stem.

The graduation of the stem of the hydrometer is made in two ways: either, so as to indicate directly the specific gravity, or by an arbitrary division into degrees, from which the specific gravity must be found by calculation.

The hydrometers in most frequent use are, in this country, Sikes'; in France, Germany, &c., those of Beaumè and of Beck. The hydrometers of Gay-Lussac and Cartier are less generally employed.

The following tables indicate the relations that exist between the indications afforded by the different kinds of hydrometers, and between the densities and per-centage value of various liquids.

I. TABLE for ascertaining the density of liquids heavier than water, from the hydrometer degrees of Beaumè and of Beck.

Degrees.	Beaumè at 14°·4 Cels.	Beck.	Degrees.	Beaumè at 14°·4 Cels.	Beck.
0	1·000	1·0000	39	1·336	1·2977
1	1·007	1·0059	40	1·347	1·3077
2	1·013	1·0119	41	1·359	1·3178
3	1·020	1·0180	42	1·371	1·3281
4	1·027	1·0241	43	1·384	1·3386
5	1·033	1·0303	44	1·396	1·3492
6	1·040	1·0366	45	1·408	1·3600
7	1·047	1·0429	46		1·3710
8	1·055	1·0494	47		1·3821
9	1·062	1·0559	48		1·3934
10	1·069	1·0625	49		1·4050
11	1·077	1·0692	50		1·4167
12	1·084	1·0759	51		1·4286
13	1·092	1·0828	52		1·4407
14	1·099	1·0897	53		1·4530
15	1·107	1·0968	54		1·4655
16	1·115	1·1039	55		1·4783
17	1·123	1·1111	56		1·4912
18	1·132	1·1184	57		1·5044
19	1·140	1·1258	58		1·5179
20	1·148	1·1333	59		1·5315
21	1·157	1·1409	60		1·5454
22	1·166	1·1486	61		1·5596
23	1·174	1·1565	62		1·5741
24	1·183	1·1644	63		1·5888
25	1·192	1·1724	64		1·6038
26	1·201	1·1806	65		1·6190
27	1·211	1·1888	66		1·6346
28	1·220	1·1972	67		1·6505
29	1·230	1·2057	68		1·6667
30	1·239	1·2143	69		1·6832
31	1·249	1·2230	70		1·7000
32	1·260	1·2319	71		1·7172
33	1·270	1·2409	72		1·7347
34	1·281	1·2500	73		1·7526
35	1·291	1·2593	74		1·7708
36	1·302	1·2687	75		1·7895
37	1·313	1·2782	76		1·8085
38	1·325	1·2879			

II. TABLE for ascertaining the density of liquids lighter than water, from the hydrometer degrees of Beaumè and of Beck.

Degrees.	Beaumè at 12°·5 Cels.	Cartier at 12°·5 Cels.	Beck.	Degrees.	Beaumè at 12°·5 Cels.	Cartier at 12°·5 Cels.	Beck.
0			1·0000	31	0·8742	0·8707	0·8457
1			0·9941	32	0·8690	0·8652	0·8415
2			0·9883	33	0·8639	0·8598	0·8374
3			0·9826	34	0·8588	0·8545	0·8333
4			0·9770	35	0·8538	0·8491	0·8292
5			0·9714	36	0·8488	0·8439	0·8252
6			0·9659	37	0·8439	0·8387	0·8212
7			0·9604	38	0·8391	0·8336	0·8173
8			0·9550	39	0·8343	0·8286	0·8133
9			0·9497	40	0·8295		0·8095
10	1·0000		0·9444	41	0·8249		0·8061
11	0·9932		0·9392	42	0·8202		0·8018
12	0·9865		0·9340	43	0·8156		0·7981
13	0·9799		0·9289	44	0 8111		0·7944
14	0·9733	0·9764	0·9239	45	0·8066		0·7907
15	0·9669	0·9695	0·9189	46	0·8022		0·7871
16	0·9605	0·9627	0·9139	47	0·7978		0·7834
17	0·9542	0·9560	0·9090	48	0·7935		0·7799
18	0·9480	0·9493	0·9042	49	0·7892		0·7763
19	0·9420	0·9427	0·8994	50	0·7849		0·7727
20	0·9359	0·9363	0·8947	51	0·7807		0·7692
21	0·9300	0·9299	0·8900	52	0·7766		0·7658
22	0·9241	0·9237	0·8854	53	0·7725		0·7623
23	0·9183	0·9175	0·8808	54	0·7684		0·7589
24	0·9125	0·9114	0·8762	55	0·7643		0·7556
25	0·9068	0·9054	0·8717	56	0·7604		0·7522
26	0·9012	0·8994	0·8673	57	0·7565		0·7489
27	0·8957	0·8935	0·8629	58	0·7526		0·7456
28	0·8902	0·8877	0·8585	59	0·7487		0·7423
29	0·8848	0·8820	0·8542	60	0·7449		0·7391
30	0·8795	0·8763	0·8500				

III. Proportions of anhydrous sulphuric acid (SO^3) and monohydrated acid (SO^3,HO) corresponding to different densities.

Density.	Anhydrous sulphuric acid.	Monohydrated acid.	Density.	Anhydrous sulphuric acid.	Monohydrated acid.
1·842	81·5	100	1·466	47·3	58
1·841	80·1	98	1·456	46·6	57
1·840	78·8	97	1·446	45·7	56
1·839	77·5	95	1·436	44·9	55
1·837	76·3	93	1·427	44·0	54
1·836	75·4	92	1·417	43·2	53
1·834	74·1	91	1·407	42·4	52
1·832	73·8	90	1·398	41·6	51
1·831	72·5	89	1·388	40·8	50
1·828	71·2	88	1·379	40·0	49
1·822	70·4	87	1·370	39·1	48
1·814	69·6	86	1·361	38·3	47
1·796	68·8	85	1·353	37·5	46
1·788	67·9	84	1·344	36·7	45
1·777	67·1	83	1·335	35·9	44
1·769	66·3	82	1·326	35·1	43
1·757	65·5	81	1·317	34·3	42
1·747	64·7	80	1·308	33·4	41
1·736	63·9	79	1·300	32·6	40
1·725	63·1	78	1·291	31·8	39
1·712	62·3	77	1·282	31·0	38
1·699	61·4	76	1·274	30·2	37
1·685	60·6	75	1·265	29·4	36
1·675	59·8	74	1·257	28·5	35
1·663	59·0	73	1·248	27·7	34
1·652	58·2	72	1·241	26·9	33
1·642	57·4	71	1·233	26·3	32
1·632	56·6	70	1·225	25·5	31
1·620	55·7	69	1·218	24·1	30
1·609	54·9	68	1·211	23·7	29
1·598	54·1	67	1·203	22·8	28
1·587	53·3	66	1·196	22·0	27
1·576	52·5	65	1·188	21·2	26
1·565	51·6	64	1·179	20·4	25
1·550	50·8	63	1·171	19·6	24
1·539	50·0	62	1·163	18·8	23
1·528	49·2	61	1·155	17·9	22
1·517	48·4	60	1·148	17·1	21
1·507	47·6	59	1·141	16·3	20
1·496	46·7	58	1·133	15·5	19
1·486	45·9	57	1·125	14·7	18
1·476	45·1	56	1·117	13·9	17

TABLE III. (*continued*).

Density.	Anhydrous sulphuric acid.	Monohydrated acid.	Density.	Anhydrous sulphuric acid.	Monohydrated acid.
1·109	13·1	16	1·054	6·5	8
1·102	12·2	15	1·048	5·7	7
1·095	11·4	14	1·041	4·9	6
1·089	10·6	13	1·034	4·1	5
1·081	9·8	12	1·027	3·3	4
1·074	9·0	11	1·021	2·4	3
1·068	8·2	10	1·014	1·6	2
1·061	7·3	9	1·007	0·8	1

IV. PER-CENTAGE AMOUNTS of chlorine and hydrochloric acid gas corresponding to different densities of hydrochloric acid solution.

Density.	Chlorine.	Hydrochloric acid gas.	Density.	Chlorine.	Hydrochloric acid gas.
1·2000	39·675	40·777	1·1494	29·361	30·174
1·1982	39·278	40·369	1·1473	28·964	29·767
1·1964	38·882	39·961	1·1452	28·567	29·359
1·1946	38·485	39·554	1·1431	28·171	28·951
1·1928	38·089	39·146	1·1410	27·772	28·544
1·1910	37·692	38·738	1·1389	27·376	28·136
1·1893	37·296	38·330	1·1369	26·979	27·728
1·1875	36·900	37 923	1·1349	26·583	27·321
1·1857	36·503	37·516	1·1328	26·186	26·913
1·1846	36·107	37·108	1·1308	25·789	26·505
1·1822	35·707	36·700	1·1287	25·392	26·098
1·1802	35·310	36·292	1·1267	24·996	25·690
1·1782	34·913	35·884	1·1247	24·599	25·282
1·1762	34·517	35·476	1·1227	24·202	24·874
1·1741	34·121	35·068	1·1206	23·805	24·466
1·1721	33·724	34·660	1·1185	23·408	24·058
1·1701	33·328	34·252	1·1164	22·012	23·650
1·1681	32·931	33·845	1·1143	22·615	23·242
1·1661	32·535	33·437	1·1123	22·218	22·834
1·1641	32·136	33·029	1·1102	21·822	22·426
1·1620	31·746	32·621	1·1082	21·425	22·019
1·1599	31·343	32·213	1·1061	21·028	21·611
1·1578	30·946	31·805	1·1041	20·632	21·204
1·1557	30·550	31·398	1·1020	20·235	20·796
1·1537	30·153	30·090	1·1000	19·837	20·388
1·1515	29·757	30·582	1·0980	19·440	19·980

TABLE IV. (*continued*).

Density.	Chlorine.	Hydrochloric acid gas.	Density.	Chlorine.	Hydrochloric acid gas.
1·0960	19·044	19·572	1·0477	9·522	9·786
1·0939	18·647	19·165	1·0457	9·126	9·379
1·0919	18·250	18·757	1·0437	8·729	8·971
1·0899	17·854	18·349	1·0417	8·332	8·563
1·0879	17·457	17·941	1·0397	7·935	8·155
1·0859	17·060	17·534	1·0377	7·538	7·747
1·0838	16·664	17·126	1·0357	7·141	7·340
1·0818	16·267	16·718	1·0337	6·745	6·932
1·0798	15·870	16·310	1·0318	6·348	6·524
1·0778	15·474	15·902	1·0298	5·951	6·116
1·0758	15·077	15·494	1·0299	5·554	5·709
1·0738	14·680	15·087	1·0259	5·158	5·301
1·0718	14·284	14·679	1·0239	4·762	4·894
1·0697	13·887	14·271	1·0220	4·365	4·486
1·0677	13·490	13·863	1·0200	3·968	4·078
1·0657	13·094	13·457	1·0180	3·571	3·670
1·0637	12·697	13·049	1·0160	3·174	3·262
1·0617	12·300	12·641	1·0140	2·778	2·854
1·0597	11·903	12·233	1·0120	2·381	2·447
1·0577	11·506	11·825	1·0100	1·984	2·039
1·0557	11·109	11·418	1·0080	1·588	1·631
1·0537	10·712	11·010	1·0060	1·191	1·124
1·0517	10·316	10·602	1·0040	0·795	0·816
1·0497	9·919	10·194	$1·002_0$	0·397	0·408

V. PER-CENTAGE AMOUNTS of anhydrous nitric acid (NO^5) and of hydrated acid (NO^5,HO) corresponding to different densities of nitric acid.

Density.	Anhydrous acid.	Hydrated acid.	Density.	Anhydrous acid.	Hydrated acid.	Density.	Anhydrous acid.	Hydrated acid.
1·500	79·7	100	1·470	70·9	89	1·431	62·2	78
1·498	78·9	99	1·467	70·1	88	1·427	61·4	77
1·496	78·1	98	1·464	69·4	87	1·423	60·6	76
1·494	77·3	97	1·460	69·3	86	1·419	59·8	75
1·491	76·5	96	1·457	67·7	85	1·415	59·0	74
1·488	75·7	95	1·453	66·9	84	1·411	58·2	73
1·485	74·9	94	1·450	66·1	83	1·406	57·4	72
1·482	74·1	93	1·446	65·3	82	1·402	56·5	71
1·479	73·3	92	1·442	64·5	81	1·398	55·8	70
1·476	72·5	91	1·439	63·8	80	1·394	55·0	69
1·473	71·7	90	1·435	63·0	79	1·388	54·2	68

TABLE V. (*continued*).

Density.	Anhydrous acid.	Hydrated acid.	Density.	Anhydrous acid.	Hydrated acid.	Density.	Anhydrous acid.	Hydrated acid.
1·383	53·4	67	1·258	35·1	44	1·123	17·5	22
1·378	52·6	66	1·252	34·3	43	1·117	16·7	21
1·373	51·8	65	1·246	33·5	42	1·111	15·9	20
1·368	51·1	64	1·240	32·7	41	1·105	15·1	19
1·363	50·2	63	1·234	31·9	40	1·099	14·3	18
1·358	49·4	62	1·228	31·1	39	1·093	13·5	17
1·353	48·6	61	1·221	30·3	38	1·088	12·7	16
1·348	47·8	60	1·215	29·5	37	1·082	11·9	15
1·343	47·0	59	1·208	28·7	36	1·076	11·2	14
1·338	46·2	58	1·202	27·9	35	1·071	10·4	13
1·332	45·4	57	1·196	27·1	34	1·065	9·6	12
1·327	44·6	56	1·189	26·3	33	1·059	8·8	11
1·322	43·8	55	1·183	25·5	32	1·054	8·0	10
1·316	43·0	54	1·177	24·7	31	1·048	7·2	9
1·311	42·2	53	1·171	23·9	30	1·043	6·4	8
1·306	41·4	52	1·165	23·1	29	1·037	5·6	7
1·300	40·6	51	1·159	22·3	28	1·032	4·8	6
1·295	39·8	50	1·153	21·5	27	1·027	4·0	5
1·289	39·0	49	1·146	20·7	26	1·021	3·2	4
1·283	38·3	48	1·140	19·9	25	1·016	2·4	3
1·276	37·5	47	1·134	19·1	24	1·011	1·6	2
1·270	36·7	46	1·129	18·3	23	1·005	0·8	1
1·264	35·9	45						

VI. PER-CENTAGE AMOUNTS of hydrated acid ($C^4H^4O^4$) corresponding to different densities of acetic acid.

Anhydrous acid.	Density at 60° F.	Anhydrous acid.	Density at 60° F.	Anhydrous acid.	Density at 60° F.
0	1·0000	14	1·0245	28	1·0460
1	1·0019	15	1·0261	29	1·0472
2	1·0037	16	1·0277	30	1·0485
3	1·0055	17	1·0293	31	1·0498
4	1·0072	18	1·0310	32	1·0510
5	1·0089	19	1·0326	33	1·0522
6	1·0107	20	1·0342	34	1·0537
7	1·0124	21	1·0358	35	1·0546
8	1·0141	22	1·0373	36	1·0558
9	1·0159	23	1·0389	37	1·0569
10	1·0177	24	1·0404	38	1·0580
11	1·0194	25	1·0419	39	1·0591
12	1·0211	26	1·0433	40	1·0601
13	1·0228	27	1·0447	41	1·0611

TABLE VI. (*continued*).

Anhydrous acid.	Density at 60° F.	Anhydrous acid.	Density at 60° F.	Anhydrous acid.	Density at 60 F.
42	1·0621	57	1·0735	72	1·0759
43	1·0631	58	1·0740	73	1·0754
44	1·0640	59	1·0745	74	1·0748
45	1·0649	60	1·0749	75	1·0741
46	1·0658	61	1·0753	76	1·0732
47	1·0667	62	1·0756	77	1·0722
48	1·0675	63	1·0759	78	1·0710
49	1·0683	64	1·0762	79	1·0696
50	1·0691	65	1·0764	80	1·0681
51	1·0698	66	1·0765	81	1·0664
52	1·0705	67	1·0766	82	1·0646
53	1·0711	68	1·0766	83	1·0626
54	1·0717	69	1·0766	84	1·0603
55	1·0723	70	1·0765	85	1·0574
56	1·0729	71	1·0763	85·11	1·0570

VII. PER-CENTAGE AMOUNTS of ammonia (NH^3) corresponding to different densities of solution of ammonia.

Density.	Per-centage.	Density.	Per-centage.	Density.	Per-centage.
0·8750	32·50	0·9326	17·52	0·9573	18·82
0·8875	29·25	0·9385	15·88	0·9597	10·17
0·9000	26·00	0·9435	14·53	0·9619	9·60
0·9054	25·37	0·9476	13·46	0·9692	9·50
0·9166	22·07	0·9513	12·40		
0·9254	19·54	0·9545	11·56		

VIII. PER-CENTAGE AMOUNTS of anhydrous potash (KO) corresponding to different densities of solution of potash.

Density.	Per-centage.	Density.	Per-centage.	Density.	Per-centage.
1·3300	28·290	1·1979	18·671	1·0819	8·487
1·3131	27·158	1·1838	17·540	1·0703	7·355
1·2966	26·027	1·1702	16·408	1·0589	6·224
1·2805	24·895	1·1568	15·277	1·0478	5·002
1·2648	23·764	1·1437	14·145	1·0369	3·961
1·2493	22·632	1·1308	13·013	1·0260	2·829
1·2342	21·500	1·1182	11·882	1·0153	1·697
1·2268	20·935	1·1059	10·750	1·0050	0·5658
1·2122	19·803	1·0938	9·619		

IX. Per-centage Amounts of anhydrous soda (NaO) corresponding to different densities of solution of soda.

Density.	Per-centage.	Density.	Per-centage.	Density.	Per-centage.
1·4285	30·220	1·2912	19·945	1·1330	9·066
1·4193	29·616	1·2843	19·341	1·1233	8·462
1·4101	29·011	1·2775	18·730	1·1137	7·857
1·4011	28·407	1·2708	17·132	1·1042	7·253
1·3923	27·802	1·2642	17·528	1·0948	6·648
1·3836	27·200	1·2578	16·923	1·0855	6·944
1·3751	26·594	1·2515	16·319	1·0464	5·540
1·3668	25·989	1·2453	15·814	1·0675	4·835
1·3586	25·385	1·2392	15·110	1·0587	4·231
1·3505	24·780	1·2280	14·506	1·0500	3·626
1·3426	24·176	1·2178	13·901	1·0414	3·022
1·3349	23·572	1·2058	13·297	1·0330	2·418
1·3273	22·967	1·1948	12·692	1·0246	1·813
1·3198	22·363	1·1841	12·088	1·0163	1·209
1·3143	21·884	1·1734	11·484	1·0081	0·604
1·3125	21·894	1·1630	10·879	1·0040	0·302
1·3053	21·154	1·1528	10·275		
1·2982	20·550	1·1428	9·670		

X. Per-centage Amounts of anhydrous carbonate of soda corresponding to different densities of solution of soda.

Density.	Per-centage.	Density.	Per-centage.	Density.	Per-centage.
1·4812	40·504	1·3177	26·432	1·1282	11·748
1·4750	40·139	1·3078	25·454	1·1166	10·769
1·4626	39·160	1·2980	24·475	1·1052	9·760
1·4504	38·181	1·2836	23·496	1·0940	8·811
1·4384	37·202	1·2694	22·517	1·0829	7·832
1·4265	36·223	1·2554	21·538	1·0719	6·853
1·4147	35·244	1·2417	20·539	1·0611	5·874
1·4030	34·265	1·2282	19·580	1·0505	4·895
1·3915	33·286	1·2150	18·601	1·0401	3·916
1·3808	32·307	1·2020	17·622	1·0299	2·934
1·3692	31·328	1·1892	16·643	1·0108	1·958
1·3585	30·349	1·1766	15·664	1·0098	0·979
1·3480	29·360	1·1642	14·685	1·0048	0·489
1·3378	28·391	1·1520	13·706		
1·3277	27·412	1·1400	12·727		

TABLE XI. PER-CENTAGE AMOUNTS, by weight and volume, of absolute alcohol, corresponding to different densities of diluted spirit.

Density.	Alcohol by weight.	Alcohol by volume.	Strength.	Hydrometer indication.
{ 7938 F { 7946	100·00	100·00 }	Absolute alcohol.	
7969 F	99·00			
7995	....	99·00		
8001 F	98·00			
8031 F	97·00			
8041	....	98·00		
8061 F	96·00			
8084	....	97·00		
8089 F	95·00	97·61		
8118 F	94·00			
8125	....	96·00		
8145 F	93·00			
8156	....	....	67·0	0°
8160	....	....	66·8	0·2
8163	....	....	66·6	0·4
8164	92·46	95·00		
8167	....	....	66·5	0·6
8170	....	....	66·3	0·8
8172 F	92·00			
8174	....	....	66·1	1·0
8178	....	....	65·9	1·2
8181	....	....	65·8	1·4
8185	....	....	65·6	1·6
8188	....	....	65·5	1·8
8192	....	....	65·3	2·0
8196	....	....	65·1	2·2
8199 F	91·00	....	65·0	2·4
8201	....	94·00		
8203	....	....	64·8	2·6
8206	....	....	64·7	2·8
8210	....	....	64·5	3·0
8214	....	....	64·3	3·2
8218	....	....	64·1	3·4
8221	....	....	64·0	3·6
8224	....	....	63·8	3·8
8227				
8228 F	90·00	94·46	63·6	4·0
8231	....	....	63·4	4·2

The densities marked F, and the corresponding per-centages of alcohol by weight, are those estimated by Fownes.

TABLE XI. (*continued*).

Density.	Alcohol by weight.	Alcohol by volume.	Strength.	Hydrometer indication.
8234	....	....	63·2	4·4
8238	....	93·00		
8237	....	....	63·1	4·6
8242	....	....	62·9	4·8
8245	....	....	62·7	5·0
8249	....	....	62·5	5·2
8252	....	....	62·3	5·4
8254 F	89·00			
8256	....	....	62·2	5·6
8259	....	....	62·0	5·8
8263	....	....	61·8	6·0
8266	....	....	61·6	6·2
8270	....	....	61·4	6·4
8270		....		
8272	....			
8273	....	92·00	61·3	6·6
8277	....	....	61·1	6·8
8279 F	88·00			
8280	....	....	60·9	7·0
8284	....	....	60·7	7·2
8287	....	....	60·5	7·4
8291	....	....	60·4	7·6
8294	....	....	60·2	7·8
8298	....	....	60·0	8·0
8301	....	....	59·8	8·2
8305 F	87·00	....	59·6	8·4
8306	....	91·00		
8308	....	....	59·5	8·6
8312	....	....	59·3	8·8
8315	....	....	59·1	9·0
8319	....	....	58·9	9·2
8322	....	....	58·7	9·4
8326	....	....	58·6	9·6
8329	....	....	58·4	9·8
8331 F	86·00			
8333	....	....	58·2	10·0
8336	....	....	58·0	10·2
8339	85·75	90·00		
8340	....	....	57·8	10·4
8344	....	....	57·7	10·6
8347	....	....	57·5	10·8
8351	....	....	57·3	11·0
8354	....	....	57·1	11·2
8358	....	....	56·9	11·4

TABLE XI. (*continued*).

Density.	Alcohol by weight.	Alcohol by volume.	Strength.	Hydrometer indication.
8357 F	85·00	90·88		
8362	....	....	56·8	11·6
8365	....	....	56·6	11·8
8369	....	....	56·4	12·0
8372	....	....	56·2	12·2
8373	....	89·00		
8376	....	....	56·0	12·4
8379	....	....	55·9	12·6
8382 F	84·00			
8383	....	....	55·7	12·8
8386	....	....	55·5	13·0
8390	....	....	55·3	13·2
8393	....	....	55·1	13·4
8396	....	....	55·0	13·6
8400	....	....	54·8	13·8
8403	....	....	54·6	14·0
8405	....	88·00		
8407	....	....	54·4	14·2
8408 F	83·00			
8410	....	....	54·2	14·4
8413	....	....	54·1	14·6
8417	....	....	53·9	14·8
8420	....	....	53·7	15·0
8424	....	....	53·5	15·2
8427	....	....	53·3	15·4
8431	....	....	53·1	15·6
8434 F	82·00	....	52·9	15·8
8436	....	87·00		
8438	....	....	52·7	16·0
8441	....	....	52·5	16·2
8445	....	....	52·3	16·4
8448	....	....	52·1	16·6
8452	....	....	51·9	16·8
8455	....	....	51·7	17·0
8459 F	81·00	....	51·5	17·2
8462	....	....	51·3	17·4
8465	....	....	51·1	17·6
8466	....	86·00		
8469	....	....	50·9	17·8
8472	....	....	50·7	18·0
8476	....	....	50·5	18·2
8480	....	....	50·3	18·4
8482	....	....	50·1	18·6
8483 F	80·00	86·97		

TABLE XI. (*continued*).

Density.	Alcohol by weight.	Alcohol by volume.	Strength.	Hydrometer indication.
8486	....	....	49·9	18·8
8490	....	....	49·7	19·0
8493	....	....	49·5	19·2
8496	79·50	85·00	49·3	19·4
8499	....	....	49·1	19·6
8503	....	....	48·9	19·8
8506	....	....	48·7	20·0
8508 F	79·00			
8510	....	....	48·5	20·2
8513	....	....	48·3	20·4
8516	....	....	48·0	20·6
8520	....	....	47·8	20·8
8523	....	....	47·6	21·0
8526	....	84·00		
8527	....	....	47·4	21·2
8530	....	....	47·2	21·4
8533 F	78·00	....	47·0	21·6
8537	....	....	46·8	21·8
8540	....	....	46·6	22·0
8543	....	....	46·4	22·2
8547	....	....	46·2	22·4
8550	....	....	46·0	22·6
8553	....	....	45·8	22·8
8555	....	83·00		
8556	....	....	45·6	23·0
8557 F	77·00			
8560	....	....	45·4	23·2
8563	....	....	45·2	23·4
8566	....	....	45·0	23·6
8570	....	....	44·8	23·8
8573	....	....	44·6	24·0
8577	....	....	44·4	24·2
8580	....	....	44·2	24·4
8581 F	76·00			
8583	....	82·00	43·9	24·6
8587	....	....	43·7	24·8
8590	....	....	43·5	25·0
8594	....	....	43·3	25·2
8597	....	....	43·1	25·4
8601	....	....	42·8	25·6
8604	....	....	42·6	25·8
8603 F	75·00	82·80		
8608	....	....	42·4	26·0
8611	....	81·00	42·2	26·2

TABLE XI. (*continued*).

Density.	Alcohol by weight.	Alcohol by volume.	Strength.	Hydrometer indication.
8615	....	....	42·0	26·4
8618	....	....	41·7	26·6
8622	....	....	41·5	26·8
8625 F	74·00	....	41·3	27·0
8629	....	....	41·1	27·2
8632	....	....	40·9	27·4
8636	....	....	40·6	27·6
8639	73·59	80·00	40·4	27·8
8643	....	....	40·2	28·0
8646	....	....	40·0	28·2
8649 F	73·00			
8650	....	....	39·8	28·4
8653	....	....	39·5	28·6
8657	....	....	39·3	28·8
8660	....	....	39·1	29·0
8664	....	....	38·9	29·2
8665	....	79·00		
8667	....	....	38·7	29·4
8671	....	....	38·4	29·6
8672 F	72·00			
8674	....	....	38·2	29·8
8778	....	....	38·0	30·0
8681	....	....	37·8	30·2
8685	....	....	37·6	30·4
8688	....	....	37·3	30·6
8692	....	....	37·1	30·8
8693	....	78·00		
8695	....	....	36·9	31·0
8696 F	71·00			
8699	....	....	36·7	31·2
8702	....	....	36·4	31·4
8706	....	....	36·2	31·6
8709	....	....	35·9	31·8
8713	....	....	35·7	32·0
8716	....	....	35·5	32·2
8720	....	77·00	35·2	32·4
8721 F	70·00	78·40	35·0	32·6
8723				
8727	....	....	34·7	32·8
8730	....	....	34·5	33·0
8734	....	....	34·3	33·2
8737	....	....	34·1	33·4
8741	....	....	33·8	33·6
8744	....	....	33·6	33·8

TABLE XI. (*continued*).

Density.	Alcohol by weight.	Alcohol by volume.	Strength.	Hydrometer indication.
8745 F	69·00			
8747	....	76·00		
8748	....	....	33·4	34·0
8751	....	....	33·2	34·2
8755	....	....	32·9	34·4
8758	....	....	32·7	34·6
8762	....	....	32·4	34·8
8765	....	....	32·2	35·0
8769 F	68·00	....	32·0	35·2
8772	....	....	31·7	35·4
8773	67·93	75·00		
8776	....	....	31·5	35·6
8779	....	....	31·2	35·8
8783	....	....	31·0	36·0
8786	....	....	30·8	36·2
8790	....	....	30·5	36·4
8793 F	67·00	....	30·3	36·6
8797	....	....	30·0	36·8
8799	....	74·00		
8800	....	....	29·8	37·0
8804	....	....	29·5	37·2
8807	....	....	29·3	37·4
8811	....	....	29·0	37·6
8814	....	....	28·8	37·8
8816 F	66·00			
8818	....	....	28·5	38·0
8822	....	....	28·3	38·2
8825	....	73·00	28·0	38·4
8829	....	....	27·8	38·6
8832	....	....	27·5	38·8
8836	....	....	27·3	39·0
8840 F	65·00	73·79	27·0	39·2
8843	....	....	26·8	39·4
8847	....	....	26·5	39·6
8850	....	72·00	26·3	39·8
8854	....	....	26·0	40·0
8858	....	....	25·8	40·2
8861	....	....	25·5	40·4
8863 F	64·00			
8865	....	....	25·3	40·6
8869	....	....	25·0	40·8
8872	....	....	24·8	41·0
8875	....	71·00		
8876	....	....	24·5	41·2

TABLE XI. (*continued*).

Density.	Alcohol by weight.	Alcohol by volume.	Strength.	Hydrometer indication.
8879	....	....	24·3	41·4
8883	....	....	24·0	41·6
8886 F	63·00	....	23·8	41·8
8890	....	....	23·5	42·0
8894	....	....	23·2	42·2
8897	....	....	23·0	42·4
8900	62·51	70·00		
8901	....	....	22·7	42·6
8904	....	....	22·5	42·8
8908 F	62·00	....	22·2	43·0
8912	....	....	21·9	43·2
8915	....	....	21·7	43·4
8919	....	....	21·4	43·6
8922	....	....	21·2	43·8
8925				
8926	....	69·00	20·9	44·0
8930	....	....	20·6	44·2
8932 F	61·00			
8933	....	....	20·4	44·4
8937	....	....	20.1	44·6
8940	....	....	19·9	44·8
8944	....	....	19·6	45·0
8948	....	....	19·3	45·2
8949				
8951	....	68·00	19·1	45·4
8955	....	....	18·8	45·6
8956 F	60·00	68·97		
8959	....	....	18·6	45·8
8963	....	....	18·3	46·0
8966	....	....	18·0	46·2
8970	....	....	17·7	46·4
8973	....	67·00		
8974	....	....	17·5	46·6
8977	....	....	17·2	46·8
8979 F	59·00			
8981	....	....	16·9	47·0
8985	....	....	16·6	47.2
8989	....	....	16·4	47·4
8992	....	....	16·1	47·6
8996	....	....	15·9	47·8
8997	....	66·00		
9000	....	....	15·6	48·0
9001 F	58·00	....		
9004	....	....	15·3	48·2

TABLE XI. (*continued*).

Density.	Alcohol by weight.	Alcohol by volume.	Strength.	Hydrometer indication.
9008	....	....	15·0	48·4
9011	....	....	14·8	48·6
9015	....	....	14·5	48·8
9019	....	....	14·2	49·0
9021	57·25	65·00		
9023	....	....	13·9	49·2
9025 F	57·00			
9026	....	....	13·6	49·4
9030	....	....	13·4	49·6
9034	....	....	13·1	49·8
9038	....	....	12·8	50·0
9041	....	....	12·5	50·2
9044	....	64·00		
9045	....	....	12·2	50·4
9047 F	56.00			
9049	....	....	12·0	50·6
9052	....	....	11·7	50·8
9056	....	....	11·4	51·0
9060	....	....	11·1	51·2
9064	....	....	10·8	51·4
9067	....	63·00	10·6	51·6
9069 F	55·00	63·97		
9071	....	...	10·3	51·8
9075	....	....	10·0	52·0
9079	....	....	9·7	52·2
9082	....	....	9·4	52·4
9085	....	....	9·2	52·6
9089	....	....	8·9	52·8
9090 F	54·00	62·00		
9093	....	....	8·6	53·0
9097	....	....	8·3	53·2
9100	....	....	8·0	53·4
9104	....	....	7·7	53·6
9107	....	....	7·4	53·8
9111	....	....	7·1	54·0
9112	....	61·00		
9113 F	53·00			
9115	....	....	6·8	54·2
9118	....	....	6·5	54·4
9122	....	....	6·2	54·6
9126	....	....	5·9	54·8
9130	....	....	5·6	55·0
9134	52·20	60·00	5·3	55·2
9135 F	52·00			

TABLE XI. (*continued*).

Density.	Alcohol by weight.	Alcohol by volume.	Strength.	Hydrometer indication.
9137	....	....	5·0	55·4
9141	....	....	4·8	55·6
9145	....	....	4·5	55·8
9148	....	....	4·2	56·0
9152	....	....	3·9	56·2
9156	....	59·00	3·6	56·4
9159	....	....	3·3	56·6
9162 F	51·00			
9163	....	....	3·0	56·8
9166	....	....	2·7	57·0
9170	....	....	2·4	57·2
9174	....	....	2·1	57·4
9178	....	58·00	1·9	57·6
9182	....	....	1·6	57·8
9184 F	50·00	58·79		
9185	....	....	1·3	58·0
9189	....	....	1·0	58·2
9192	....	....	0·7	58·4
9196	....	....	0·3	58·6
9198	49·24	57·05	Proof spirit.	58·8
9200	....	57·00		
9204	....	....	0·3	59·0
9206 F	49·00			
9207	....	....	0·6	59·2
9210	....	....	0·9	59·4
9214	....	....	1·3	59·6
9218	....	....	1·6	59·8
9221	....	56·00		
9222	....	....	1·9	60·0
9226	....	....	2·2	60·2
9228 F	48·00			
9229	....	....	2·5	60·4
9233	....	....	2·8	60·6
9237	....	....	3·1	60·8
9241	....	....	3·4	61·0
9242	47·29	55·00		
9244	....	....	3·7	61·2
9248	47·00	....	4·0	61·4
9252	....	....	4·4	61·6
9255	....	....	4·7	61·8
9259	....	....	5·0	62·0
9263	....	54·00	5·3	62·2

TABLE XI. (*continued*).

Density.	Alcohol by weight.	Alcohol by volume.	Strength.	Hydrometer indication.
9267	....	....	5·7	62·4
9270	46·00	....	6·0	62·6
9274	....	....	6·4	62·8
9278	....	....	6·7	63·0
9282	....	....	7·0	63·2
9283	....	53·00		
9286	....	....	7·3	63·4
9291 F	45·00	53·43	7·7	63·6
9295	....	....	8·0	63·8
9399	....	....	8·3	64·0
9302	....	....	8·6	64·2
9303	....	52·00		
9306	....	....	9·0	64·4
9310	....	....	9·3	64·6
9314 F	44·00	....	9·7	64·8
9318	....	....	10·0	65·0
9322	....	....	10·0	65·0
9323	....	51·00	10·3	65·2
9326	....	.....	10·7	65·4
9329	....	....	10·0	65·6
9333	....	....	11·4	65·8
9335 F	43·00			
9337	....	....	11·7	66·0
9341	....	....	12·1	66·2
9343	42·52	50·00		
9345	....	....	12·4	66·4
9349	....	....	12·8	66·6
9353	....	....	13·1	66·8
9357 F	42·00	....	13·5	67·0
9360	....	....	13·9	67·2
9362	....	49·00		
9364	....	....	14·2	67·4
6368	....	....	14·6	67·6
9372	....	....	14·9	67·8
9376 F	41·00	....	15·3	68·0
9380	....	....	15·7	68·2
9381	....	48·00		
9384	....	....	16·0	68·4
9388	....	....	16·4	68·6
9392	....	....	16·7	68·8
9396 F	40·00	47·92	17·1	69·0
9399	....	47·00	17·5	69·2
9403	....	....	17·8	69·4
9407	....	....	18·2	69·6

TABLE XI. (*continued*).

Density.	Alcohol by weight.	Alcohol by volume.	Strength.	Hydrometer indication.
9411	....	....	18·5	69·8
9415	....	....	18·9	70·0
9417 F	39·00	46·00		
9419	....	....	19·3	70·2
9422	....	....	19·7	70·4
9426	....	....	20·0	70·6
9430	....	....	20·4	70·8
9434 F	38·00	....	20·8	71·0
9435	37·90	45·00		
9437	....	....	21·2	71·2
9441	....	....	21·6	71·4
9445	....	....	21·9	71·6
9448	....	....	22·3	71·8
9452 F	37·00	44·00	22·7	72·0
9456	....	....	23·1	72·2
9460	....	....	23·5	72·4
9464	....	....	23·9	72·6
9468	....	....	24·3	72·8
9470 F	36·00	43·00		
9472	....	....	24·7	73·0
9476	....	....	25·1	73·2
9480	....	....	25·5	73·4
9484	....	....	25·9	73·6
9487	....	42·00		
9488	....	....	26·3	73·8
9490 F	35·00	42·25		
9492	....	....	26·7	74·0
9496	....	....	27·1	74·2
9499	....	....	27·5	74·4
9503	....	41·00	28·0	74·6
9507	....	....	28·4	74·8
9511 F	34·00	....	28·4	75·0
9515	....	....	29·2	75·2
9519	33·39	40·00	29·7	75·4
9522	....	....	30·1	75·6
9526	....	....	30·6	75·8
9528 F	33·00			
9530	....	....	31·0	76·0
9534	....	....	31·4	76·2
9535	....	39.00		
9538	....	....	31·9	76·4
9542	....	....	32·3	76·6
9544 F	32·00			
9546	....	....	32·8	76·8

TABLE XI. (*continued*).

Density.	Alcohol by weight.	Alcohol by volume.	Strength.	Hydrometer indication.
9550	....	38·00	33·2	77·0
9553	....	....	33·7	77·2
9557	....	....	34·2	77·4
9561 F	31·00	....	34·6	77·6
9565	....	37·00	35·1	77·8
9567	....	....	35·6	78·0
9573	....	....	36·1	78·2
9577 F	30·00	36·45	36·6	78·4
9579	....	36·00		
9580	....	....	37·1	78·6
9584	....	....	37·6	78·8
9588	....	....	38·1	79·0
9592	....	....	38·6	79·2
9593 F	29·00			
9596	28·99	35·00	39·1	79·4
9599	....	....	39·6	79·6
9603	....	....	40·1	79·8
9605	....	34·00		
9607	....	....	40·6	80·0
9609 F	28·00			
9611	....	....	41·1	80·2
9615	....	....	41·7	80·4
9618	....	33·00		
9619	....	....	42·2	80·6
9623 F	27·00	....	42·8	80·8
9627	....	....	43·3	81·0
9631	....	32·00	43·9	81·2
9635	....	....	44·4	81·4
9638 F	26·00	....	45·0	81·6
9642	....	....	45·5	81·8
9643	....	31·00		
9646	....	....	46·1	82·0
9650	....	....	46·7	82·2
9652 F	25·00	30·55		
9654	....	....	47·3	82·4
9655	24·69	30·00		
9657	....	....	47·9	82·6
9661	....	....	48·5	82·8
9665 F	24·00	....	49·1	83·0
9666	....	29·00		
9669	....	....	49·7	83·2
9673	....	....	50·3	83·4
9677 F	23·00	28·00	51·0	83·6
9681	....	....	51·6	83·8

TABLE XI. (*continued*).

Density.	Alcohol by weight.	Alcohol by volume.	Strength.	Hydrometer indication.
9685	....	....	52·2	84·0
9688	....	27·00		
9689	....	....	52·9	84·2
9691 F	22·00			
9693	....	....	53·5	84·4
9697	....	....	54·2	84·6
9698	....	26·00		
9701	....	....	54·8	84·8
9705 F	21·00	....	55·5	85·0
9709	20·46	25·00	56·2	85·2
9713	....	....	56·9	85·4
9716 F	20·00	24·57		
9718	....	....	57·6	85·6
9719	....	24·00		
9722	....	....	58·3	85·8
9726	....	....	59·0	86·0
9729 F	19·00	23·00		
9730	....	....	59·7	86.2
9734	....	....	60·4	86·4
9738	....	....	61·1	86·6
9740 F	18·00	22·00		
9742	....	....	61·8	86·8
9746	....	....	62·5	87·0
9750	....	21·00	63·2	87·2
9754 F	17·00	....	63·9	87·4
9758	....	....	64·6	87·6
9760	16·28	20·00		
9762	....	....	65·3	87·8
9766 F	16·00	....	66·0	88·0
9770	....	19·00	66.7	88·2
9774	....	....	67·4	88·4
9778 F	15·00	18·52	68·0	88·6
9780	....	18·00		
9782	....	....	68·7	88·8
9786	....	....	69·4	89·0
9790 F	14·00	17·00	70·1	89·2
9794	....	....	70·8	89·4
9798	....	....	71·4	89·6
9800	....	16·00		
9802 F	13·00	....	72·1	89·8
9806	....	....	72·8	90·0
9810	....	....	73·5	90·2
9811	12·15	15·00		
9814 F	12·00	....	74·1	90·4

TABLE XI. (*continued*).

Density.	Alcohol by weight.	Alcohol by volume.	Strength.	Hydrometer indication.
9818	....	....	74·8	90·6
9821		14·00		
9822	....	....	75·4	90·8
9826	....	....	76·1	91·0
9828 F	11·00			
9830	....	....	76·7	91·2
9832	....	13·00		
9834	....	....	77·3	91·4
9838	10·07	12·42	78·0	91·6
9839	9·99			
9840	9·92			
9841	9·85			
9842	9·78			
9843	9·70	12·00	78·6	91·8
9844	9·63			
9845	9·56			
9846	9·49	....	79·2	92·0
9847	9·41			
9848	9·34			
9849	9·27			
9850	9·20	....	79·8	92·2
9851	9·12			
9852	9·05			
9853	8·98			
9854	8·91	11·00	80·4	92·4
9855	8·84			
9856	8·77			
9857	8·70			
9858	8·63	....	81·1	92·6
9859	8·55			
9860	8·48			
9861	8·41			
9862	8·34	....	81·7	92·8
9863	8·27			
9864	8·20			
9865	8·13			
9866	8·06	10·00	82·3	93·0
9867	7·99			
9868	7·92			
9869	7·85			
9870	7·78	....	82·9	93·2
9871	7·71			
9872	7·64			
9873	7·57			

TABLE XI. (*continued*).

Density.	Alcohol by weight.	Alcohol by volume.	Strength.	Hydrometer indication.
9874	7·50	....	83·5	93·4
9875	7·43			
9876	7·37			
9877	7·30			
9878	7·23	....	84·0	93·6
9879	7·16	9·00		
9880	7·09			
9881	7·02			
9882	6·95	....	84·6	93·8
9883	6·89			
9884	6·82			
9885	6·75			
9886	6·69	....	85·2	94·0
9887	6·62			
9888	6·55			
9889	6·49			
9890	6·42			
9891	6·35	8·00	85·8	94·2
9892	6·29			
9893	6·22			
9894	6·15	....	86·3	94·4
9895	6·09			
9896	6·02			
9897	5·96			
9898	5·89	....	86·9	94·9
9899	5·83			
9900	5·77			
9901	5·70			
9902	5·64	....	87·4	94·8
9903	5·58			
9904	5·51	7·00		
9905	5·45			
9906	5·39	....	88·0	95·0
9907	5·32			
9908	5·26			
9909	5·20			
9910	5·13	....	88·5	95·2
9911	5·07			
9912	5·01	6·25		
9913	4·94			
9914	4·88	....	89·1	95·4
9915	4·82			
9916	4·76	6·00		
9917	4·70			

TABLE XI. (*continued*).

Density.	Alcohol by weight.	Alcohol by volume.	Strength.	Hydrometer indication.
9918	4·64	....	89·6	95·6
9919	4·57			
9920	4·51			
9921	4·45			
9922	4·39	....	90·2	95·8
9923	4·33			
9924	4·27			
9925	4·20			
9926	4·14	....	90·7	96·0
9927	4·08			
9928	4·02	5·00		
9929	3·96			
9930	3·90	....	91·2	96·2
9931	3·84			
9932	3·78			
9933	3·73			
9934	3·67	....	91·7	96·4
9935	3·61			
9936	3·55			
9937	3·49			
9938	3·43	....	92·3	96·6
9939	3·37			
9940	3·32			
9941	3·26			
9942	3·20	....	92·8	96·8
9943	3·15			
9944	3·08			
9945	3·02			
9946	2·97	....	93·3	97·0
9947	2·91			
9948	2·85			
9949	2·79			
9950	2·74	....	93·8	97·2
9951	2·69			
9952	2·62			
9953	2·57			
9954	2·51	....	94·3	97·4
9955	2·45			
9956	2·39			
9957	2·34			
9958	2·28	....	94·9	97·6
9959	2·22			
9960	2·17			
9961	2·11			

TABLE XI. (*continued*).

Density.	Alcohol by weight.	Alcohol by volume.	Strength.	Hydrometer indication.
9962	2·05	....	95·4	97·8
9963	1·99			
9964	1·94			
9965	1·89			
9966	1·83	....	95·9	98·0
9967	1·79			
9968	1·73			
9969	1·67			
9970	1·61	2·00	96·4	98·2
9971	1·56			
9972	1·51			
9973	1·45			
9974	1·40	....	96·8	98·4
9975	1·34			
9976	1·29			
9977	1·23			
9978	1·18	....	97·3	98·6
9979	1·12			
9980	1·07			
9981	1·02			
9982	0·96	....	97·7	98·8
9983	0·91			
9984	0·85			
9985	0·80			
9986	0·74	....	98·2	99·0
9987	0·69			
9988	0·64			
9989	0·58			
9990	0·53	....	98·7	99·2
9991	0·47			
9992	0·42			
9993	0·37	....	99·1	99·4
9994	0·32			
9995	0·26			
9996	0·21			
9997	0·16	....	99·6	99·6
9998	0·11			
9999	0·05			
10000	....	....	100·00	99·8 100·0

XII. TABLE showing the amounts of crystallizable sugar corresponding to different densities of solution of sugar; and the amounts of extract in wort or in beer deprived of alcohol.

Amount of sugar, or of malt extract.	Densities of sugar solution at 56°·2 F. *Niemann.*	Densities of beer wort. *Zennek.*	Densities of malt infusion or sugar solution. *Balling.*	Amount of sugar, or of malt extract.	Densities of sugar solution at 56°·2 F. *Niemann.*
0	1·0000			25	1·1056
1	1·0035			26	1·1103
2	1·0070			27	1·1150
3	1·0106	1·01140	1·012	28	1·1197
3·5		1·01340		29	1·1245
4	1·0143	1·01550	1·016	30	1·1293
4·5		1·01780		31	1·1340
5	1·0179	1·01970	1·020	32	1·1388
5·5		1·02190		33	1·1436
6	1·0215	1·02410	1·024	34	1·1484
6·5		1·02610		35	1·1533
7	1·0254	1·02820	1·028	36	1·1582
7·5		1·03030		37	1·1631
8	1·0291		1·032	38	1·1681
9	1·0328		1·036	39	1·1731
10	1·0367		1·040	40	1·1781
11	1·0410		1·044	41	1·1832
12	1·0426		1·048	42	1·1883
13	1·0504		1·053	43	1·1935
14	1·0552		1·057	44	1·1989
15	1·0600		1·061	45	1·2043
16	1·0647		1·065	46	1·2098
17	1·0693		1·070	47	1·2153
18	1·0738		1·074	48	1·2209
19	1·0784		1·078	49	1·2265
20	1·0830		1·083	50	1·2322
21	1·0875		1·087	51	1·2378
22	1·0920		1·092	52	1·2434
23	1·0965		1·095	53	1·2490
24	1·1010		1·101	54	1·2546

TABLE XIII. COMPARISON of the specific gravities of solutions of cane-sugar, starch-sugar, pale and brown malt, dextrin, caramel from cane-sugar, and of the extract substance produced in the fermentation of sugar, all containing equal amounts of carbon.

Graham, *Hofmann*, and *Redwood*.

Cane-sugar in 1000 parts by weight.	Carbon in 1000 parts by weight.	Specific gravity of solution of cane-sugar.	Specific gravity of solution of starch-sugar.	Solution of pale malt.	Solution of brown malt.	Solution of dextrin.	Solution of caramel.	Solution of extractive substance.
25	10·53	1010·1	1010·4	1010·0	1010·0	1009·7	1008·7	1008·9
50	21·05	1020·2	1020·8	1020·3	1020·2	1019·3	1017·3	1017·8
75	31·58	1030·2	1031·3	1030·6	1030·6	1028·8	1026·2	1026·5
100	42·10	1040·6	1042·4	1041·2	1041·2	1038·3	1034·9	1035·5
125	52·63	1051·0	1053·5	1052·1	1052·0	1047·9	1043·8	1044·7
150	63·16	1061·8	1064·9	1063·0	1062·9	1057·3	1052·8	1053·9
175	73·68	1072·9	1076·0	1074·2	1074·0	1066·9	1062·3	1063·0
200	84·21	1083·8	1087·8	1085·5	1085·5	1067·6	1071·8	1072·7
225	94·73	1095·2	1099·4	1097·2	1097·2	1086·3	1081·3	1082·3
250	105·26	1106·7	1111·4	1109·0	1109·0	1095·8		

TABLE XIV. Equivalent weights of the Elements.

	Symbol.	O = 100.	H = 1.
Aluminum	Al	342·5	27·4
Antimony	Sb	1612·5	129
Arsenic	As	937·5	75
Barium	Ba	856·25	68·5
Bismuth	Bi	2600	208
Boron	B	136·25	10·9
Bromine	Br	1000	80
Cadmium	Cd	700	56
Calcium	Ca	250	20
Carbon	C	75	6
Chlorine	Cl	443·75	35·5
Chromium	Cr	333·75	26·7
Cobalt	Co	368·75	29·5
Copper	Cu	396·25	31·7
Fluorine	F	237·5	19
Gold	Au	2462·5	197
Hydrogen	H	12·5	1
Iodine	I	1588·75	127·1
Iron	Fe	350	28
Lead	Pb	1296·25	103·7
Magnesium	Mg	150	12
Manganese	Mn	345	27·6
Mercury	Hg	1250	100
Molybdenum	Mo	575	46
Nickel	Ni	370	29·6
Nitrogen	N	175	14
Oxygen	O	100	8
Palladium	Pd	666·25	53·3
Phosphorus	P	387·5	31
Platinum	Pt	1233·75	98·7
Potassium	K	490	39·2
Silicium	Si	266·25	21·3
Silver	Ag	1351·25	108·1
Sodium	Na	287·5	23
Strontium	Sr	547·5	43·8
Sulphur	S	200	16
Tin	Sn	725	58
Uranium	U	750	60
Zinc	Zn	407·5	32·6

TABLE XV. RELATION of the scales of Celsius, Réaumur, and Fahrenheit.

Celsius.	Réaumur.	Fahrenheit.	Celsius.	Réaumur.	Fahrenheit.	Celsius.	Réaumur.	Fahrenheit.
+100	+80	+212	+53	+42·4	+127·4	+ 6	+ 4·8	+ 42·8
99	79·2	210·2	52	41·6	125·6	5	4	41
98	78·4	208·4	51	40·8	123·8	4	3·2	39·2
97	77·6	206·6	50	40	122	3	2·4	37·4
96	76·8	204·8	49	39·2	120·2	2	1·6	35·6
95	76	203	48	38·4	118·4	1	0·8	33·8
94	75·2	201·2	47	37·6	116·6	0	0	32
93	74·4	199·4	46	36·8	114·8	− 1	− 0·8	30·2
92	73·6	197·6	45	36	113	2	1·6	28·4
91	72·8	195·8	44	35·2	111·2	3	2·4	26·6
90	72	194	43	34·4	109·4	4	3·2	24·8
89	71·2	192·2	42	33·6	107·6	5	4	23
88	70·4	190·4	41	32·8	105·8	6	4·8	21·2
87	69·6	188·6	40	32	104	7	5·6	19·4
86	68·8	186·8	39	31·2	102·2	8	6·4	17·6
85	68	185	38	30·4	100·4	9	7·2	15·8
84	67·2	183·2	37	29·6	98·6	10	8	14
83	66·4	181·4	36	28·8	96·8	11	8·8	12·2
82	65·6	179·6	35	28	95	12	9·6	10·4
81	64·8	177·8	34	27·2	93·2	13	10·4	8·6
80	64	176	33	26·4	91·4	14	11·2	6·8
79	63·2	174·2	32	25·6	89·6	15	12	5
78	62·4	172·4	31	24·8	87·8	16	12·8	3·2
77	61·6	170·6	30	24	86	17	13·6	1·4
76	60·8	168·8	29	23·2	84·2	18	14·4	− 0·4
75	60	167	28	22·4	82·4	19	15·2	2·2
74	59·2	165·2	27	21·6	80·6	20	16	4
73	58·4	163·4	26	20·8	78·8	21	16·8	5·8
72	57·6	161·6	25	20	77	22	17·6	7·6
71	56·8	159·8	24	19·2	75·2	23	18·4	9·4
70	56	158	23	18·4	73·4	24	19·2	11·2
69	55·2	156·2	22	17·6	71·6	25	20	13
68	54·4	154·4	21	16·8	69·8	26	20·8	14·8
67	53·6	152·6	20	16	68	27	21·6	16·6
66	52·8	150·8	19	15·2	66·2	28	22·4	18·4
65	52	149	18	14·4	64·4	29	23·2	20·2
64	51·2	147·2	17	13·6	62·6	30	24	22
63	50·4	145·4	16	12·8	60·8	31	24·8	23·8
62	49·6	143·6	15	12	59	32	25·6	25·6
61	48·8	141·8	14	11·2	57·2	33	26·4	27·4
60	48	140	13	10·4	55·4	34	27·2	29·2
59	47·2	138·2	12	9·6	53·6	35	28	31
58	46·4	136·4	11	8·8	51·8	36	28·8	32·8
57	45·6	134·6	10	8	50	37	29·6	34·6
56	44·8	132·8	9	7·2	48	38	30·4	36·4
55	44	131	8	6·4	46	39	31·2	38·2
54	43·2	129·2	7	5·6	44·6	40	32	40

TABLE XVI. Showing the relation between the weights of different countries.

English avoirdupois pound.	French kilo-gramme.	Austrian and Bavarian pound.	Hanove-rian pound.	Hessian pound.	Swiss, Baden, and Hesse Darmstadt pound.	Prussian pound.	Saxon pound.	Brunswick and Würtem-berg pound.	Altenburg pound.
1	0·4540	0·8100	0·9260	0·9360	0·9070	0·9700	0·9700	0·9710	0·9720
2·2050	1	1·7860	2·0430	2·0660	2·0000	2·1380	2·1390	2·1400	2·1440
1·2340	0·5600	1	1·1440	1·1570	1·0170	1·1974	1·1976	1·1198	1·2003
1·0786	0·4896	0·8742	1	1·0112	0·9790	1·0567	1·0470	1·0475	1·0495
1·0668	0·4841	0·8663	0·9686	1	0·9681	1·0350	1·0354	1·0355	1·0735
1·0378	0·5000	0·8930	1·0125	1·0330	1	1·0692	1·0695	1·0700	1·0720
1·0310	0·4677	0·8352	0·9553	0·9661	0·9582	1	1·0002	1·0007	1·0050
1·0307	0·4674	0·8350	0·9551	0·9659	0·9350	0·9998	1	1·0005	1·0024
1·0301	0·4673	0·8346	0·9547	0·9654	0·9346	0·9993	0·9996	1	1·0023
1·0258	0·4665	0·8330	0·9529	0·9636	0·9328	0·9974	0·9977	0·9981	1

French measures of weight.	English grains.	Troy ounces = 480 grains.	Avoirdupois lbs. = 7000 grains.	Cwts. = 112 lbs. = 784000 grains.	Tons = 20 cwts. 15680000 grs.
Milligramme	0·015432	0·000032	0·0000022	0·00000002	0·00000001
Centigramme	0·154323	0·000322	0·0000220	0·00000020	0·00000010
Decigramme	1·543235	0·003215	0·0002205	0·00000197	0·00000009
Gramme	15·432349	0·032151	0·0022046	0·00001968	0·00000984
Decagramme	154·323488	0·321507	0·0220462	0·00019684	0·00000842
Hectogramme	1543·234880	3·215073	0·2204621	0·00196841	0·00009842
Kilogramme	15432·348800	32·150727	2·2046213	0·01968412	0·00098420
Myriogramme	154323·488000	321·507267	22·0462126	0·19684118	0·00984205

1 grain = 0·064798950 gramme. 1 troy oz. = 31·103496 grm. 1 lb. avdp. = 0·45359265 kilogr. 1 cwt. = 50·8023709 kilogrs.

French measures of capacity.	In cubic inches.	In cubic feet = 1728 cubic inches.	In pints = 34·65923 cubic inches.	In gallons = 8 pints = 277·27384 cubic inches.	In bushels = 8 gallons = 2218·19075 cubic inches.
Millilitre, or cubic centimètre	0·061027	0·0000353	0·001761	0·00022010	0·00002752
Centilitre, or 10 cubic centimètres	0·610271	0·0003532	0·017608	0·00220097	0·00027512
Decilitre, or 100 cubic centimètres	6·102705	0·0035317	0·176077	0·02200967	0·00275120
Litre, or cubic décimètre	61·027052	0·0353166	1·760773	0·22009668	0·02751208
Decalitre, or centistère	610·270515	0·3531658	17·607734	2·20096677	0·27512084
Hectolitre, or decistère	6102·705152	3·5316581	176·077341	22·00966767	2·75120845
Kilolitre, or stère, or cubic mètre	61027·051519	35·3165807	1760·773414	220·09667675	27·51208459
Myriolitre, or decastère	610270·515194	353·1658074	17607·734140	2200·96676750	275·12084593

1 cubic inch = 16·3861759 cubic centimètres. 1 cubic foot = 28·3153119 cubic décimètres. 1 gallon = 4·543457969 litres.

French measures of length.	English inches.	English feet = 12 inches.	English yards = 3 feet.	English fathoms = 6 feet.	English miles = 1760 yards.
Millimètre	0·03937	0·0032809	0·0010936	0·0005468	0·0000006
Centimètre	0·39371	0·0328090	0·0109363	0·0054682	0·0000062
Décimètre	3·93708	0·3280899	0·1093633	0·0546816	0·0000621
Mètre	39·37079	3·2808992	1·0936331	0·5468165	0·0006214
Décamètre	393·70790	32·8089920	10·9363310	5·4681655	0·0062138
Hectomètre	3937·07900	328·0899200	109·3633100	54·6816550	0·0621382
Kilomètre	39370·79000	3280·8992000	1093·6331000	546·8165500	0·6213824
Myriomètre	393707·90000	32808·9920000	10936·3310000	5468·1655000	6·2138244

English measures of weight.

Troy weight.

Pound.	Ounces.	Dwts.	Grains.
		1	24
	1	20	480
1	12	240	5760

Avoirdupois weight.

Ton.	Cwt.	Pounds.	Ounces.	Drachms.	Grains.
			1	16	437½
		1	16	....	7000
	1	112	1792		
1	20	2240	35840		

Apothecaries weight.

Pound.	℥ Ounces.	ʒ Drachms.	℈ Scruples.	gr. Grains.
			1	20
		1	3	60
	1	8	24	480
1	12	96	288	5760

The grain is the same in all of these weights.

English measures of capacity.

Dry measure.

Load or Wey.	Quarters.	Bushels.	Pecks.	Gallons.
			1	2
		1	4	8
	1	8	32	64
1	5	40	160	320

The imperial gallon contains 277·274 cubic inches. This quantity of pure water at 62° F., under an atmospheric pressure of 30 inches, weighs 10 pounds avoirdupois or 70·000 grains. A cubic inch of water weighs 252·458 grains.

Liquid measure.

	Barrel.	Kilderkin.	Firkin.	Gallons.	Quarts.	Pints.	Gills.
						½	1
					½	1	4
					1	2	
				1	4	8	
			1	9	36	72	
		1	2	18	72	144	
	1	2	4	36	144	288	
Hogshead of ale ...	....	....	....	54	216	432	
„ wine	....	....	....	63	252	504	
Puncheon.....	....	....	....	84	336	672	
Butt of ale	....	....	....	108	432	864	
Pipe=2 Hogsheads	....	....	....	126	504	1008	
Tun=2 Pipes	....	....	....	252	1008	2016	

Cubic measure.

Yard.	Foot.	Inch.
	1	1728
1	27	46656

The bulk of a barrel is 5 cubic feet.

Linear measure.

Mile.	Furlong.	Chain.	Pole.	Yard.	Foot.	Inch.
					1	12
				1	3	36
			1	5½	16½	198
		1	4	22	66	792
	1	10	40	220	660	7920
1	8	80	320	1760	5280	63360

The length of a pendulum that vibrates seconds of mean time in the latitude of London, at the level of the sea, and in a vacuum, is 39·1393 inches.

END.

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